

EFFECT OF VARIETY, STORAGE AND RIPENESS OF APPLES
(*Malus domestica* Borkh.) ON PHYSICAL AND CHEMICAL PARAMETERS
AFFECTING APPLESAUCE RHEOLOGICAL PROPERTIES

A Dissertation

Presented to the Faculty of the Graduate School

of Cornell University

In Partial Fulfillment of the Requirements for the Degree of

Doctor of Philosophy

by

Luciana Pereira e Ferreira

May 2013

© 2013 Luciana Pereira e Ferreira

EFFECT OF VARIETY, STORAGE AND RIPENESS OF APPLES
(*Malus domestica* Borkh.) ON PHYSICAL AND CHEMICAL PARAMETERS
AFFECTING APPLESAUCE RHEOLOGICAL PROPERTIES

Luciana Pereira e Ferreira, Ph.D.
Cornell University, 2013

Over 3 harvest years (2009, 2010 and 2011), rheological properties of applesauce in relation to fruit ripening and sauce physical and chemical parameters were assessed. Ten varieties were used to obtain single-variety applesauce (Ben Davis, Cortland, Crispin, Empire, Golden Delicious, Idared, Jonagold, McIntosh, Rhode Island Greening, Rome Beauty) by hot and cold-break methods. The effect of storage and fruit ripening were studied using different post-harvest conditions:

- Cold storage (1-4 °C at 95% relative humidity) for up to 8 months. Sauce was prepared monthly for collection of at least 5 experimental points.
- Varying storage temperatures for assessing the benefit of controlled post-harvest fruit ripening – 10 and 21 °C for up to 30 days. Sauce was prepared every 3-7 days for collection of at least 5 experimental points.
- Controlled atmosphere (CA) storage (1-3% O₂ and 1-4% CO₂ at 1-4 °C). Apples out of CA storage were held at 10 °C for up to 35 days. Sauce was prepared every 3-7 days for collection of at least 5 experimental points and results were compared to those of freshly harvested apples subjected to controlled post-harvest fruit ripening.

Apples were evaluated for ripeness (firmness, pH, acidity, soluble solids); and applesauce for rheology (USDA consistency, yield stress, consistency index and serum capillary viscosity) and physical and chemical parameters – particle size distribution (PSD), mean

particle size (MPS) and particle size distribution span (PSDS); moisture, calcium, starch, alcohol insoluble residue (AIR), total soluble pectin (TSP) and pectin degree of methoxylation (PDM). Results were analyzed by ANOVA and significant differences among means determined by Tukey's test ($p \leq 0.05$). Harvest year, variety, storage condition, fruit ripeness and their interaction were significant factors for sauce consistency. Differences in rheological parameters are explained by differences in MPS (500-1200 μm) and PSDS (0.9-2.25); starch (0.01-0.78%), AIR (1.5-5.5%) and TSP (0.11-0.75%) contents; and PDM (33-95%). Best consistency applesauce was achieved with smaller MPS ($\leq 800 \mu\text{m}$), larger PSDS (≥ 1.5) and/or higher AIR ($\geq 2.5\%$), TSP ($\geq 0.25\%$) and lower PDM ($\leq 60\%$). Differences in chemical parameters of sauce between harvest years might be related to weather conditions affecting apple composition.

BIOGRAPHICAL SKETCH

Luciana Ferreira was born on June 21st 1986 in Belém, in the Amazon Region of Brazil. During high school, she ran her own chocolate and confectionery business, Chocoluci. She obtained her bachelor's degree in Food Engineering at Universidade Federal do Pará in 2009. While pursuing her undergraduate degree she was a scholar for 2 years for the National Council of Technological and Scientific Development (CNPQ), when she carried out research focusing on functional properties of Amazon fruits and plant extracts. In her Junior year in 2008, she participated in the Cornell Summer Scholars Program under the orientation of Dr. Olga Padilla-Zakour. In 2009 she was accepted as a graduate student at Cornell University. During her time at Cornell University, she engaged in diverse extra-curricular activities including LUBRASA – The Luso-Brazilian Student Association at Cornell University; SAGES – The Student Association of the Geneva Experiment Station; and a number of product development teams, among which she was a co-leader for the Developing Solutions for Developing Countries IFTSA winning team in 2010 with the MandiMais project. She was also a founder of CHOCTECH – Cornell University Chocolate and Confections Technology Club. Luciana intends to pursue the academic career working at her hometown in Amazon, a Region where much is to be discovered and developed within the field of Food Science, and to be an enthusiast for the research, entrepreneurship and valorization of Amazonian raw materials.

To God, source of all blessings;

To my parents, Ana Celeste Pereira Ferreira and Octávio José Pessoa Ferreira,

For the gift of life, and for everything I am;

To my brother, Rodrigo Octávio, for being such a good example to follow;

To my fiancé, Alex Pinheiro Centeno, for his unconditional love and support;

To Grampa Earl and the Wheelers, for having such a big positive impact in my life.

ACKNOWLEDGMENTS

My sincere gratitude to Dr. Olga Padilla-Zakour for giving me the opportunity of being here; for her guidance and support during the length of the program and for allowing me to explore my own interests, making it a true Cornell Experience.

Special thanks my committee members, Dr. Randy Worobo and Dr. Miguel Gomez for valuable contributions to this work; as well as to Dr. Harry Lawless who I had the pleasure of working for as a teaching assistant in 2009; Dr. Dennis Miller and Dr. Syed Rizvi for their support to our Developing Solutions for Developing Countries team in 2010; and Dr. Gavin Sacks, Dr. Joe Regenstein and Dr. Rao for their support to CHOCTECH.

Many thanks to my lab mate, Nongnuch Athiphunamphai and to our lab technician, Herbert Cooley, who helped me in all aspects of this research and without whom everything would have been much harder.

Further thanks to our pilot plant manager, Thomas Gibson, and also to Cheryl Leach and Elizabeth Sullivan, who additionally helped in the applesauce project; as well as to John Churey and Dr. David Manns for their time and assistance with research equipment and methodology implementation.

To my colleagues and friends in Ithaca and Geneva: you made the graduate school experience much more enjoyable and fun with your presence, friendship and shared love for food. I hope we can always keep in touch and gather for other potlucks in the future!

To The Dr. Pepper Snapple Group; The Graduate School; The Department of Food Science; The Feeding Tomorrow Foundation of IFT; The Western New York IFT

and The Juice Products Association: thank you so much for the financial support during the course of my study.

I also would like to recognize the importance of the Wheeler family in my journey. Sue, Tom and Morgan: you touched my life in ways I can never be thankful enough. Thank you so much for making me feel part of the family as well.

Finally, to my beloved family, mom, dad, Digo and Alex: you are everything to me, my greatest inspiration. Without you I could never have gone this far. Thank you so much for always being there for me!

TABLE OF CONTENTS

	PAGE
BIOGRAPHICAL SKETCH	iii
DEDICATION	iv
ACKNOWLEDGEMENTS	v
TABLE OF CONTENTS	vii
LIST OF FIGURES	ix
LIST OF TABLES	xi
CHAPTER 1: BACKGROUND AND LITERATURE REVIEW	1
Background	2
Literature Review	6
References	13
CHAPTER 2: APPLE VARIETY AND REFRIGERATED STORAGE EFFECTS ON CONSISTENCY OF HOT-BREAK APPLESAUCE	18
Abstract and Keywords	19
Practical Application	20
Introduction	20
Materials and Methods	22
Results and Discussion	27
Conclusions	39
References	40
CHAPTER 3: EFFECT OF VARIETY AND RIPENESS OF APPLES (<i>Malus domestica</i> Borkh.) ON PHYSICAL AND CHEMICAL PARAMETERS AFFECTING RHEOLOGICAL PROPERTIES OF COLD-BREAK APPLESAUCE	47
Abstract and Keywords	48
Practical Application	49
Introduction	49
Materials and Methods	52
Results and Discussion	56
Conclusions	71
References	72
CHAPTER 4: COLD-BREAK APPLESAUCE RHEOLOGICAL PROPERTIES IMPROVE WITH CONTROLLED POST-HARVEST FRUIT RIPENING	78
Abstract and Keywords	79
Practical Application	80
Introduction	80
Materials and Methods	82
Results and Discussion	86
Conclusions	103
References	103

TABLE OF CONTENTS

	PAGE
CHAPTER 5: CONSISTENCY OF COLD-BREAK APPLESAUCE MADE FROM CONTROLLED ATMOSPHERE STORED APPLES (<i>Malus domestica</i> Borkh.)	108
Abstract and Keywords	109
Practical Application	110
Introduction	110
Materials and Methods	113
Results and Discussion	117
Conclusions	130
References	130
CHAPTER 6: CONCLUSIONS, FUTURE WORK AND RECOMMENDATIONS	135
Summary of Findings	136
Future Work	139
APPENDIX	142

LIST OF FIGURES

FIGURE		PAGE
2.1	Hot-break applesauce processing diagram.	23
2.2	USDA consistency (sauce and free-liquid flow) of applesauce made monthly from apples stored at 1 °C and 95% relative humidity over 8 months of post-harvest storage time – (a) thicker sauce varieties; (b) thinner sauce varieties.	28
2.3	Firmness of apples stored at 1 °C and 95% relative humidity over 8 months of post-harvest storage time – (a) thicker sauce varieties; (b) thinner sauce varieties.	30
2.4	Correlation between USDA consistency (sauce flow) and yield stress of applesauce made monthly from apples stored at 1 °C and 95% relative humidity over 8 months of post-harvest storage time (n =320).	32
2.5	Capillary viscosity of serum collected from applesauce made monthly from apples stored at 1 °C and 95% relative humidity over 8 months of fruit post-harvest storage time – (a) thicker sauce varieties; (b) thinner sauce varieties.	33
2.6	Particle size distribution of applesauce made monthly from apples stored at 1 °C and 95% relative humidity (CS) over 4 months of fruit post-harvest storage time (months 2-6 in CS) – (a) thicker sauce varieties; (b) thinner sauce varieties.	34
2.7	Orthographic 3D Scatterplot of L, a and b color parameters values of applesauce made monthly from apples stored at 1 °C and 95% relative humidity (CS) over 4 months of fruit post-harvest storage time (months 2-6 in CS).	39
3.1	Firmness of apples stored at 1 °C and 95% relative humidity over 5 months of storage, over 2 harvest years.	57
3.2	USDA consistency (sauce and free-liquid flow) of applesauce made monthly from apples stored at 1 °C and 95% relative humidity over 5 months of storage, over 2 harvest years.	58
3.3	Consistency index of applesauce made monthly from apples harvested in 2010 and 2011 stored at 1 °C and 95% relative humidity (CS) over 5 months of post-harvest storage.	62
3.4	Changes in particle size distribution of applesauce made from apples stored at 1 °C and 95% relative humidity over 5 months of apple storage time (ST), over 2 harvest years.	66
3.5	Changes in starch content of applesauce made monthly from apples stored at 1 °C and 95% relative humidity over 5 months of storage time, over 2 harvest years.	68
4.1	Firmness of apples stored at 95% relative humidity (RH) at 10 °C and at 1 °C for 4 weeks or 5 months, respectively, over 2 harvest years.	87
4.2	USDA consistency (sauce and free-liquid flow) of applesauce made from apples stored at 95% relative humidity (RH) at 10 °C and at 1 °C for 4 weeks or 5 months, respectively, over 2 harvest years.	89

LIST OF FIGURES

FIGURE		PAGE
4.3	Consistency index of applesauce made from apples stored at 95% relative humidity (RH) at 10°C and 1°C for 4 weeks or 5 months, respectively, over 2 harvest years.	91
4.4	Changes in particle size distribution of applesauce made from apples stored at 95% relative humidity (RH) at 10 °C and at 1 °C for 3 weeks or 3 months, respectively, over 2 harvest years.	95
5.1	Firmness of apples stored at 95% relative humidity (RH) at 10 °C for 4 or 5 weeks immediately after harvest and after coming out of controlled atmosphere storage (CAS) – 1-4 °C, 1-3% O ₂ and 1-4% CO ₂ for 7-10 months –, respectively, over 2 harvest years.	118
5.2	USDA consistency (sauce and free-liquid flow) of applesauce made from apples stored at 95% relative humidity (RH) at 10 °C for 4 or 5 weeks immediately after harvest and after coming out of controlled atmosphere storage (CAS) – 1-4 °C, 1-3% O ₂ and 1-4% CO ₂ for 7-10 months –, respectively, over 2 harvest years.	120
5.3	Consistency index of applesauce made from apples stored at 95% relative humidity (RH) at 10 °C for 4 or 5 weeks immediately after harvest and after coming out of controlled atmosphere storage (CAS) – 1-4 °C, 1-3% O ₂ and 1-4% CO ₂ for 7-10 months –, respectively, over 2 harvest years.	123
5.4	Changes in particle size distribution of applesauce made from apples stored at 95% relative humidity (RH) at 10 °C for 4 weeks immediately after harvest and after coming out of controlled atmosphere storage (CAS) – 1-4 °C, 1-3% O ₂ and 1-4% CO ₂ for 7-10 months – over 2 harvest years.	125

LIST OF TABLES

TABLE		PAGE
2.1	Changes in chemical parameters of applesauce made from apples stored at 1 °C and 95% relative humidity (CS) over 4 months of fruit storage (2-6 months in CS): alcohol insoluble residue (AIR), total soluble pectin (TSP) and pectin degree of methoxylation (PDM).	37
3.1	Physical and chemical parameters affecting rheological properties of applesauce.	61
3.2	Physical and chemical parameters affecting applesauce rheological properties: mean particle size (MPS); particle size distribution span (PSDS); alcohol insoluble residue (AIR); total soluble pectin (TSP) and pectin degree of methoxylation (PDM) of sauce made from apples harvested in 2010 and 2011 stored at 1 °C and 95% relative humidity over 5 months.	65
4.1	Chemical parameters affecting applesauce rheological properties: alcohol insoluble residue (AIR); total soluble pectin (TSP) and pectin degree of methoxylation (PDM) of sauce made from apples stored at 1 °C and 95% relative humidity (RH) for 5 months, over 2 harvest years.	102
5.1	Changes in alcohol insoluble residue (AIR), total soluble pectin (TSP) and pectin degree of methoxylation (PDM) of applesauce made from fresh apples and those stored at 95% relative humidity (RH) at 10 °C for 4 weeks after coming out of controlled atmosphere storage (CAS) – 1-4 °C, 1-3% O ₂ and 1-4% CO ₂ for 7-10 months – over 2 harvest years.	129

CHAPTER 1:
BACKGROUND AND LITERATURE REVIEW

1. BACKGROUND

Applesauce is a typical American product. Traditionally prepared at home following recipes brought by European immigrants, it is nowadays the predominant apple-based canned product in the United States (U.S. Apple Association, 2011), and is widely available commercially as a formulated product in family size and single serve units as well as squeezable pouches proper for on-the-go eating.

Industrial food products based on apple, such as applesauce, are often presented as healthy and convenient alternatives to candy and snacks eaten between meals, especially in the case of children (Colin-Henrion and others, 2009). This health-promoting status enjoyed by apple products is in fine tune with Americans' growing awareness of the connection between diet and health, which has given a sales boost to fruit-based prepared foods (Mintel, 2009). With a growing demand for such products, apple processors will have to be ready to meet increasingly higher consumer expectations.

Applesauce is defined by FDA (2012 – A.1 Appendix) as the food prepared from comminuted or chopped apples (*Malus domestica* Borkhausen) and it is graded according to The Grading Manual for Canned Applesauce (USDA, 2009 – A.2 Appendix) through the assessment of 5 attributes: color, flavor, absence of defects, finish, and consistency. From those, quality control of product consistency is particularly important because, according to applesauce manufacturers, consumer complaints of commercial products are often related to liquid-separation or thin sauce. Furthermore, thin consistency can pose challenges during manufacturing at the filler step, causing sauce to overflow the primary

package prior to capping or sealing, which can lead to considerable financial losses for the industry.

Traditional applesauce processing followed a hot-break procedure in which apples were peeled, cored, sliced and steam-blanching prior to finishing, formulation adjustments and pasteurization. More recently, applesauce manufacturers have been adopting a more efficient and cost-effective cold-break procedure in which sauce is obtained by direct comminuting of fruit through a turbo extractor, eliminating the energy and time demanding cooking step. Commercial product characterization of cold-break applesauce conducted by our research group has shown that applesauce rheological parameters vary greatly throughout the processing year, possibly due to changes occurring to raw materials during storage, as apples are climacteric fruits and continue to ripen after being harvested (Burg, 2004).

In an effort to reduce product variability over time, the applesauce industry has adopted practices such as varietal blending in product formulations to even out the effect of fruit composition (Wiley and Binkley, 1989); different raw material storage conditions for the availability of fruit throughout the processing year and for minimizing composition changes within storage time (Louis and Massey, 1989); as well as adjustments to the processing line when necessary.

Regarding varietal blending, a typical applesauce formulation will be made from a blend of 3-7 apple varieties out of about 20 total varieties employed by the manufacturer. The choice of varieties used by the manufacturer depends on local raw material commercial production capacity, which must suit the industry's processing volume; while the choice of varieties and their proportion in a specific blend is based on fruit ripening

indicators and empirical knowledge of varietal contribution to the overall quality of applesauce. Exact varietal contribution is difficult to estimate due to the combined effect on the resulting blend. Moreover, individual varietal contribution might be affected by fruit ripening over commercial storage conditions as well as by changes in fruit composition based on different harvest seasons.

Regarding raw material storage, two conditions are most commonly applied by the industry: cold (CS) and controlled atmosphere (CA) storages. Both are raw material storage conditions at 1-4 °C and 95-98% relative humidity, which reduce ethylene synthesis and the respiratory rate of the fruit dramatically (Patchen, 1971; Meheriuk, 1985). Under CA storage, however, there is further reduction due to the use of reduced oxygen (1-3%) and increased CO₂ (1-5%) levels in the storage atmosphere (Chu, 1992; Graell and Recasens, 1992; Siddiqui and others, 1996; Vanoli and others, 2009). As a result, while fruit can be kept for 6-9 months under CS, it can be kept for 9-12 months in CA, depending on variety.

According to communication with cold-break applesauce manufacturers, challenges to process products of optimal consistency are typically faced at the beginning of the processing year (related to the beginning of the apple harvest season which usually starts in the Northeast in September and October depending on variety), when freshly harvested apples are used as raw materials, as well as when CA stored fruit starts being sourced due to depleted supply of cold stored fruit.

Objectives

Considerations above have given rise to a few research questions:

- Do apple variety and post-harvest fruit ripening affect rheological parameters of applesauce, including consistency?
- Are freshly harvested apples associated with sauce of inferior rheological properties? And, if so, can we find post-harvest ripening conditions to accelerate potential desirable changes occurring to fruit for the improvement of product consistency made in the beginning of the harvest year?
- Finally, do CA stored apples produce sauce of similar rheological properties to freshly harvested ones? And, if so, can we find post-harvest ripening conditions to accelerate potential desirable changes occurring to apples coming out of CA storage for the improvement of consistency of products made from these apples?

The aim of this study was, thus, to be able to answer those questions by assessing the impact of fruit variety, ripening within commercial storage practices, and that of harvest season in rheological properties of applesauce, with a focus on sauce consistency. Understanding parameters that affect applesauce rheological properties may assist the industry with raw material management for the development of products of optimal quality over the processing year.

2. LITERATURE REVIEW

2.1. Applesauce Consistency

According to the USDA Grading Manual (USDA, 2009), the flow of a Grade A consistency regular style (comminuted) applesauce shall not exceed 6.5 cm whilst any free-liquid shall not exceed 0.7 cm. For Grade B, the flow of product shall not exceed 8.5 cm whilst any free-liquid shall not exceed 1 cm. Substandard (SSTD) applesauce fails to meet Grade B requirements.

Toldby and Willey (1962) studied the lyophoresis, or the liquid-solid separation in applesauce, as well as possible compositional causes for the phenomena and reported that viscosity of the free-liquid, pH and relative average particle size of the applesauce were strongly correlated with lyophoresis, while the viscosity of the free-liquid was strongly correlated with pH, relative average particle size, pectin and starch content of the sauce. The methodology used by the authors to assess and define lyophoresis, however, is not compatible with USDA specifications for applesauce consistency grading.

Usiak and others (1995) studied the effect of blanch temperature and time on rheological parameters of hot-break applesauce, including USDA consistency, of two apple varieties stored under CS conditions over 5 storage months. Statistically significant differences in product consistency observed between treatments were attributed to optimal temperature for pectin methyl-esterase (PME) activity while lower sauce flow with progress of storage was suggested to be due to changes in soluble pectin content of sauces.

Drake and others (1979) compared the quality, including USDA consistency, of hot-break applesauce made from Golden Delicious apples after 5 months of CA and CS storage. No significant difference was found in sauce consistency between treatments.

Additional literature focuses on other rheological properties of applesauce. Rao and others (1986) reported that apple cultivar, fruit firmness (related to post-harvest fruit ripening), and processing parameters significantly affected the rheology of hot-break applesauce. Qiu and Rao (1988) reported on the effect of pulp content and particle size on the yield stress of hot-break applesauce.

As stated, all previous works cited focused on product obtained by hot-break procedures. Additionally, the relationship between product consistency, as defined by USDA, and other rheological measurements is unknown. Potential correlations between the measurements could be useful to applesauce manufacturers to facilitate routine product quality control.

2.2. Apple and sauce composition in relation to rheological properties

Given that applesauce consistency as established by USDA depends on product and liquid flow – which in turn depend on factors influencing the viscosity and water holding capacity of the structure – it is believed that some factors might be major contributors to explaining the consistency behavior, including: rheology of the sauce; the amount of starch, given the thickening properties of the starch molecule (Mason, 2009); the size of particles, due to implications in the yield stress of sauce (Qiu and Rao, 1988); and pectin content along with other possible factors affecting its gelation. Because of low amounts of sugars in the matrix (9-13 °Brix), the kind of pectin coagulation most likely

to take place will be the one described by Rees and others (1982), who have proposed a mechanism to describe calcium-induced coagulation and gelation of pectin, known as the egg-box model, in which calcium ions interact with oxygen from carboxylic groups of two adjacent chains giving rise to cross-linking of the chains. The process and the strength of the resulting gels are dependent on the degree of esterification of pectin, pH, and calcium and soluble solids content (Pilgrim and others, 1991; Stephen, 1995). The actual contribution of each of the parameters mentioned to applesauce consistency has not been established.

2.2.1. Carbohydrates

a) Starch: Starch is deposited in the apple fruit very early in its development for the storage of energy. As the fruit matures, starch begins to hydrolyze into sugars. The decrease in starch begins a few weeks prior to harvest and the hydrolysis into sugars happens quite rapidly during ripening. Disappearance of starch is measured qualitatively using an iodine-potassium iodide solution spray as an indicator of fruit maturity and criterion for harvesting. Small amounts of starch can be left upon harvesting, or none at all (Smock and Neubert, 1950).

b) Pectic Substances: Pectic substances is a group designation for colloidal carbohydrate derivatives composed of chains of d-galacturonic acids found in the middle lamella and cell walls of the apple flesh, which contribute to the adhesion between cells and to the mechanical strength of the cell wall, behaving in the manner of stabilized gels (Jarvis, 1984). Protopectin is the term used for water insoluble precursors of pectins, which are formed upon protopectin hydrolysis, and are water-soluble colloidal polygalacturonic acids containing a proportion of methyl groups. If

low in methoxyl (LM) content, these acids are capable of forming gels with calcium ions at low soluble solids content (Kertesz, 1952).

c) Sugars: La Belle (1981) observed the range of soluble solids in apples to be 10-15 °Brix. The importance of sugars naturally occurring in the apples to the focus of this study is related to their possible impact on the viscosity of solutions, and on the strength of pectin-based gels.

2.2.2. Calcium:

Minerals are requisite for normal plant tissue metabolism. Calcium is present in apples associated with the cell-wall middle lamella (Smock and Neubert, 1950). Calcium in apples, apple juice and pulp is reported to range 2-13 mg/100 g or 20-130 mg/L (ppm) on a fresh weight basis (Perring, 1974; Nour and others, 2010). LM Pectins require a minimum calcium concentration in order to yield gels with desirable properties (Voragen and others, 2003).

2.2.3. Acids

The acid in apples is mainly malic, ranging between 0.2-0.8 g/100 g depending on variety and ripeness. Acidity determines, in part, eating quality of the fruit and degradation of acids after harvest causes an increase in pH (Smock and Neubert 1950), which can affect product quality.

2.3. Parameters Affecting Apple Composition

2.3.1. Variety

According to Slaterry and others (2011), of the nearly over 8000 varieties of apples known around the world, only about 100 of them are grown in commercial scale in the U.S. Apple varieties can be distinguished in many ways such as color, shape, size,

skin toughness, flesh texture, sweetness, juiciness and taste. They also vary in their appropriateness for different end uses such as fresh-eating, home-cooking, food processing or industrial uses depending on their unique characteristics (O'Rourke, 1984).

New York is the second largest apple producer in the U.S. On average, 53% of the State's production is utilized as fresh fruit while 47% is utilized for processing. It is estimated that about 38% of processed fruits are destined into apple juice and apple cider production; 47% into canned products production – including applesauce, apple slices and pie filling; 10% into production of frozen slices; and 5% are used for the production of various apple products such as vinegar, jelly, apple butter, mincemeat, and dried products. The top 10 varieties in descending order of production volume are: McIntosh, Empire, Red Delicious, Cortland, Golden Delicious, Rome, Idared, Crispin, Paula Red, and in 10th position Gala, Jonagold and Jonamac (New York Apple Association, 2011). For this study we selected 8 apple varieties based on New York State's apple production volume: Cortland, Crispin, McIntosh, Jonagold, Empire, Rome, Idared and Golden Delicious; and 2 varieties commonly used for improvement of applesauce quality based on applesauce manufacturer information: R.I. Greening and Ben Davis.

Mohr (1973 and 1989) additionally reported that particle size distribution and resulting textural properties of applesauce were dependent on cultivar of apples used in sauce making as well as on length of fruit storage.

2.3.2. Fruit Ripeness

Composition changes occur over the storage of apples related to post-harvest fruit ripening of climacteric fruit (Burg, 2004). For the focus of this study it is important to highlight a few: water loss due to fruit transpiration; variations in the total sugar content

influenced by starch hydrolysis and fruit respiration; variations in pectic substances controlled by the hydrolysis of protopectin into soluble pectin and further into nonpectic materials; and decline of acids with consequent increase in pH. Additionally, it has been reported that, on a dry-weight basis, there are no marked variations in the mineral content of apples and that the apparent increase on a fresh-weight basis has been found due to water loss (Smock and Neubert, 1950). According to Brownleader and others (2006), fruit tissue structure and softening caused by composition and changes of fruit cell wall during fruit ripening involve cell separation and cell breakage; and breakdown of the pectic-rich middle lamella will cause cells to separate as in a mealy apple, giving a textural property approaching that of a solid comprising many single cells. In addition to the above changes, the authors also reported that flavor and color changes also accompany fruit ripening. Optimum ripening stage of fruits for the processing of applesauce products is not completely understood (Wiley and Binkley, 1989).

2.3.3. Postharvest Handling Conditions

Apples will keep their metabolic activities over storage to some extent depending on postharvest handling practices to which they are submitted. The efficacy of post-harvest handling systems is related to its ability of reducing those activities to a minimum level (Kader and others, 2002), which will in turn minimize changes and allow the availability of fruit of desired quality throughout the year. The eating quality of apples has been linked to firmness, soluble solids content and titratable acidity (Hoehn and others, 2003). In order to maintain these parameters overtime, the apple industry utilizes two main postharvest handling techniques:

a) Cold Storage: fruits are stored at 1-4 °C and 90-98% relative humidity rooms up to 24 hours after harvest to minimize transpiration losses. Storage should be as quick as possible to prevent physiological and pathological disorders and flesh softening, especially for long-term storage (Smock and Neubert, 1950; Kader and others, 1985).

b) Controlled Atmosphere (CA) Storage: storage conditions of regular controlled atmosphere vary from 1-3% O₂ and 1-5% CO₂ under controlled temperature (1-4 °C) and 95-98% relative humidity (Chu, 1992; Graell and Recasens, 1992; Siddiqui and others, 1996; Vanoli and others, 2009). Under reduced O₂ conditions and increased CO₂ conditions, ethylene synthesis and the respiratory process are minimized substantially (Smock and Neubert, 1950; Kader, 1986; Watkins, 2003), slowing the post-harvest ripening process and allowing longer periods of storage than cold storage.

In addition, according to Louis and Massey (1989), processing apples are seldom used freshly harvested: accelerated post-harvest fruit ripening is often carried out by storing fruit at higher temperatures (either directly after harvest or from refrigerated storage) to accelerate the desirable changes to better suit processing purposes. La Belle (1981) suggested that benefits of using fully matured and well-ripened apples for sauce processing are related to flavor development and textural changes.

2.3.4. Harvest Season

Composition of processing apples is not affected by variety and ripening stage alone. According to Emongor and Loughheed (1994), apple quality at harvest, response to various treatments, development of physiological disorders, and retention of fruit quality at the end of storage period are greatly affected by pre-harvest factors. Narasimham and others (1988) reported that apple maturation was found to be largely governed by

meteorological conditions, viz. the temperature and precipitation during the pre-bloom period and the first half of the post-bloom period. Smock and Neubert (1950) have listed environmental and cultural factors influencing fruit physiology and composition – among environmental factors, it is important to note the effect of light, water, temperature, springtime temperature, summer temperatures and other climatic factors such as wind and hail, soil and the fertility of the soil. Cultural factors are in regard to soil fertilization, soil management, pruning, thinning, spraying, use of rootstocks, ringing and other cultural practices. These factors will potentially affect fruit composition and therefore the composition of derived processed products.

Understanding how intrinsic raw material factors, such as variety and post-harvest fruit ripening stage, as well as exogenous factors such as post-harvest storage practices, affect the consistency of applesauce might support the applesauce industry to deliver products of improved quality year-round. Because growing season has such an important role on apple composition, the impact of seasonality should be additionally addressed. Science-base knowledge of apple varieties and their changes over storage time will provide guidelines for post-harvest handling practices that may be used for the improvement of applesauce products.

REFERENCES

- Brownleader M, Jackson P, Mobasheri A, Pantelides A, Sumar S, Trevan M, Dey P. 1999. Molecular aspects of cell wall modifications during fruit ripening. *Crit Rev Food Sci Nutr* 39(2):149-64.
- Burg SP. 2004. *Postharvest Physiology and Hypobaric Storage of Fresh Produce*. Cambridge: CABI Publishing. 654 p.

- Chu CL. 1992. Postharvest control of San Jose Scale on apples by controlled atmosphere storage. *Postharvest Biol.Technol.* 1(4):361-9.
- Colin-Henrion M, Mehinagic E, Renard CMGC, Richomme P, Jourjon F. 2009. From apple to applesauce: Processing effects on dietary fibres and cell wall polysaccharides. *Food Chem.* 117(2):254-60.
- Drake SR, Nelson JW, Powers JR. 1979. The influence of controlled atmosphere storage and processing conditions on the quality of applesauce from Golden Delicious apples. *J Am Soc Hortic Sci* 104:68-70.
- Emongor VE, Murr DP, Loughheed EC. 1994. Preharvest factors that predispose apples to superficial scald. *Postharvest Biol.Technol.* 4(4):289-300.
- FDA: 21CFR145.110 – Canned Applesauce [Internet]. Silver Spring, MD: U.S. Food and Drug Administration [Accessed 2012 Sep 17]. Available from: <http://www.accessdata.fda.gov/scripts/cdrh/cfdocs/cfcfr/CFRSearch.cfm?fr=145.110>.
- Graell J, Recasens I. 1992. Effects of ethylene removal on ‘Starking Delicious’ apple quality in controlled atmosphere storage. *Postharvest Biol.Technol.* 2(2):101-8.
- Jarvis MC. 1984. Structure and properties of pectin gels in plant cell walls. *Plant Cell Environ* 7(3):153-64.
- Hoehn E, Gasser F, Guggenbühl B, Künsch U. 2003. Efficacy of instrumental measurements for determination of minimum requirements of firmness, soluble solids, and acidity of several apple varieties in comparison to consumer expectations. *Postharvest Biol.Technol.* 27(1):27-37.
- Kader AA. 1986. Biochemical and physiological basis for effects of controlled and modified atmospheres on fruits and vegetables. *Food Technol.* 40: 99-104.
- Kader, Adel A. 2002. Postharvest biology and technology: an overview. In: A.A. Kader (ed.). *Postharvest Technology of Horticultural Crops*, University of California, Agriculture and Natural Resources Publication 3311. p 39-47.
- Kertesz ZI. 1952. The pectic substances. New York: Interscience Publishers, Inc. 628 p.
- La Belle RL. 1981. Apple quality characteristics as related to various processed products. In: R. Teranishi and H. Barrera-Benitez. *Quality of selected fruits and vegetables of North America*. ACS Symposium Series. Washington: American Chemical Society. p. 61–76.

- Louis M, Massey JR. 1989. Harvesting, Storing and Handling Processing Apples. In: Downing DL. Processed apple products. New York: Van Nostrand Reinhold. p 215-238.
- Mason WR. 2009. Starch use in foods. In: BeMiller J, Whistler R. Starch. 3rd ed. San Diego: Academic Press. p 745-95.
- Meheriuk MR. 1985. Controlled-atmosphere storage conditions for some more commonly grown apple cultivars. In Proc. Fourth Controlled-Atmosphere Res Conf. North Carolina State Univ. Raleigh, 395-421.
- Mintel. 2009. Fruit: Mintel Marketing Report.
- Mohr WP. 1973. Applesauce grain. J Texture Stud 4(2):263-8.
- Mohr WP. 1989. Influence of cultivar, fruit maturity, and fruit anatomy on apple sauce particle size and texture. Int J Food Sci Technol 24(4): 403-413.
- Narasimham P, Dhanaraj S, Krishnaprakash MS, Arvindaprasad B, Krishnaprasad CA, Habibunnisa S, Ananthakrishna SM. 1988. Effect of meteorological factors on fruit maturation and the prediction of optimum harvest for apples. Scientia Horticulturae 35(3-4):217-26.
- New York Apple Association: New York apples fast facts [Internet]. Victor, NY: New York Apple Association [Accessed 2011 Jun 13]. Available from: <http://www.nyapplecountry.com/fastfacts.htm>.
- Nour V, Trandafir I, Ionica ME. 2010. Compositional characteristics of fruits of several apple (*Malus domestica* Borkh.) cultivars. Not Bot Hort Agrobot Cluj 38 (3): 228-233.
- O'Rourke AD. 1994. The world apple market. CRC, New York. 237p.
- Patchen GO. 1971. Storage of Apples and Pears. Marketing Res Rep. 924. U.S. Dept. of Agriculture.
- Perring MA. 1974. The chemical composition of apples. XI. An extraction technique suitable for the rapid determination of calcium, but not potassium and magnesium, in the fruit. J Sci Food Agric 25(3): 237-245.
- Pilgrim GW, Walter RH, Oakenfull DG. 1993. Jams, jellies and preserves. In: Walter RH. The Chemistry and Technology of Pectin. San Diego: Academic Press. p 23-50.
- Qiu C, Rao MA. 1988. Role of pulp content and particle size in yield stress of apple sauce. J Food Sci 53(4):1165-1170.

- Rao MA, Cooley HJ, Nogueira JN, McLellan MR. 1986. Rheology of apple sauce: effect of apple cultivar, firmness, and processing parameters. *J Food Sci* 51(1):176-179.
- Rees DA, Morris ER, Thom D, Madden JK. 1982. Shapes and interactions of carbohydrate chains. In: Aspinall GO. *The Polysaccharides*. Academic Press, New York. p 195-290.
- Siddiqui S, Brackmann A, Streif J, Bangerth F. 1996. Controlled atmosphere storage of apples: cell wall composition and fruit softening. *J Hortic Sci* 71(4): 613-620.
- Slaterry E, Livingston M, Greene C, Klonsky K. 2011. Characteristics of Conventional and Organic Apple Production in The United States. A Report from the Economic Research Service. United States Department of Agriculture – USDA 2011. FTS 347-01. Available at: www.ers.usda.gov. Accessed at: July 28, 2011 at 9:45 p.m.
- Smock RM, Neubert AM. 1950. Apples and apple products. New York: Interscience Publishers. P 486.
- Stephen AM. 1995. Food polysaccharides and their applications. CRC press, New York. 752 p.
- Toldby V, Willey R. 1962. Liquid-solids separation, a problem in processed applesauce. *J Am Soc Hortic Sci* 81:78-90.
- U.S. Apple Association: Production and Utilization Analysis 2011 [Internet]. Vienna, VA: U.S. Apple Association. [Accessed 2013 March 8]. Available from: <http://www.yvgsa.com/pdf/facts/USApple2011ProductionAnalysis.pdf>.
- USDA: Grading Manual for Canned Applesauce [Internet]. Washington, D.C.: United States Department of Agriculture [Accessed 2009 Sep 19]. Available from: <http://www.usda.gov>.
- Usiak AMG, Bourne MC, Rao MA. 1995. Blanch temperature/time effects on rheological properties of applesauce. *J.Food Sci.* 60(6):1289-1291.
- Vanoli M, Zerbini PE, Spinelli L, Torricelli A, Rizzolo A. 2009. Polyuronide content and correlation to optical properties measured by time-resolved reflectance spectroscopy in ‘Jonagored’ apples stored in normal and controlled atmosphere. *Food Chem.* 115(4):1450-7.
- Voragen A, Voragen F, Schols H, Visser R. 2003. Advances in pectin and pectinase research. Dordrecht: Kluwer Academic Publishers. 514 p.

Watkins, C.B. 2003. Principles and practices of postharvest handling and stress. In: Apples: Crop Physiology, Production and Uses. Feree, D. and I.J. Warrington (eds) Chapt. 23, CAB Pub. pp. 585-614.

Wiley RC, Binkley CR. 1989. Applesauce and other canned apple products. In: Downing DL. Processed apple products. New York: Van Nostrand Reinhold. p 215-238.

CHAPTER 2:
APPLE VARIETY AND REFRIGERATED STORAGE EFFECTS ON
CONSISTENCY OF HOT-BREAK APPLESAUCE

ABSTRACT: Ten different apple varieties (Ben Davis, Cortland, Crispin, Empire, Golden Delicious, Idared, Jonagold, McIntosh, Rhode Island Greening and Rome Beauty) were harvested and stored up to 8 months at 1 °C and 95% relative humidity (cold storage – CS). Applesauce was processed monthly following a hot-break process. Apples and their corresponding sauce were evaluated to assess the impact of fruit variety and storage time on physical and chemical parameters affecting sauce rheological properties, focusing on product consistency. Variety and storage time were significant factors ($p\text{-value} \leq 0.05$). Applesauce yield stress was correlated with sauce flow ($R^2 = 0.61$). Apples were grouped by rheological properties in thicker and thinner sauce varieties. Thicker sauce apples were overall firmer, yielding product of smaller mean particle size and higher particle size distribution span (ranging 558-836 μm and 1.5-2.2, respectively). Thinner sauce had higher moisture (ranging 84.0-89.5%) with increased sauce flow and some liquid separation with progress of storage. Further analysis of alcohol insoluble residue (AIR), total soluble pectin (TSP) and pectin degree of methoxylation (PDM) of 5 varieties representative of overall trends observed suggest liquid separation might be explained by low pectin content or its degradation over storage (ranging 0.12 – 0.74%). Starch was found at insignificant levels in apples ($\geq 0.2\%$) and calcium content in applesauce (21-26 ppm) did not significantly impact applesauce consistency. Differences in total applesauce yield and color based on apple variety are also reported.

Keywords: applesauce, consistency, apple variety, cold storage.

Practical Application: Varietal blending is employed by applesauce manufacturers throughout the processing year for the maintenance of product quality attributes at desired levels, among which consistency is an important parameter affecting product grading and consumer acceptance. Information about single-variety applesauce consistency over fruit post-harvest storage can assist management of blend selection for the manufacture of products of optimal consistency.

Introduction

Applesauce, identified by 21CFR145.110 (FDA, 2012) as the food made from comminuted or chopped apples, is the predominant apple-based canned product in the United States (New York Apple Association, 2011).

Following current industrial practices, applesauce is made year round from apples harvested from September to November, which are kept in cold storage (CS) for up to 5 to 8 months depending on variety and fruit condition at harvest, or for 6 to 12 months or more if kept in controlled atmosphere (CA) storage (Massey, 1989; Kader, 1986; Watkins, 2003; USDA, 2012). A blend of 3 to 7 varieties – out of about 20 commercially available for processing – is typically used to round-out the blend, in an effort to obtain a consistent product even though the raw material is changing due to the availability of varieties and how they ripen over storage (Louis and Massey, 1989; La Belle, 1981).

As climacteric fruits, apples undergo physical and chemical transformations after harvest, affecting their texture, color and flavor, which is detrimental for eating-quality fruit (Burg, 2004; Brownleader and others, 2006). Limited studies, however, have

addressed how postharvest ripening and storage affects processing quality of apples that are used for making applesauce.

Consistency, color, absence of defects, finish, and flavor are the five attributes determining applesauce quality grading (USDA, 2009). Most applesauce products contain sweeteners, flavorings (various fruits and cinnamon) and colorings, however, which reduce the impact of fruit variety on flavor and color of finished products. Control of product consistency throughout the processing year by applesauce manufacturers is more challenging as the attribute may be affected by fruit composition and ripening stage and directly affects product grading.

According to the Grading Manual for Canned Applesauce (USDA, 2009), the flow of a Grade A consistency regular style (comminuted) applesauce shall not exceed 6.5 cm whilst any free liquid shall not exceed 0.7 cm. For Grade B, the flow of product shall not exceed 8.5 cm whilst any free-liquid shall not exceed 1 cm. Substandard (SSTD) applesauce fails to meet Grade B requirements.

Toldby and Willey (1962) studied the liquid-solid separation in applesauce, reporting that it adversely affects consumer acceptance. The authors observed that apple variety and storage time significantly affect liquid-solid separation in hot-break applesauce. The methodology used by the authors to assess and define liquid separation however, does not follow USDA guidelines for applesauce consistency grading.

Way and McLellan (1989) pointed out that, commercially, some apple varieties are preferred for sauce making for their good flavor, bright color, and grain that allows for variability in particle size. Particle characteristics such as average size and distribution are important for applesauce texture and are dependent on apple variety,

ripening stage and processing parameters (Lee et al, 1965; Mohr, 1973 and 1989; Nogueira and others, 1985). Qiu and Rao (1988) reported the effect of particle mean diameter on yield stress of applesauce. The effect of particle size to applesauce consistency is not known.

Rao and others (1986) reported that apple cultivar, fruit firmness and processing parameters significantly influenced the rheology of applesauce. The relationship between applesauce consistency, as established by USDA, and other rheological properties that could help routine quality control by manufacturers is also unexplored.

No comprehensive work reports the effect of apple variety and post-harvest fruit ripening over storage on applesauce consistency. We studied rheological properties, and physical-chemical composition of applesauce made from 10 different apple varieties, commonly used for sauce making, over 8 months of CS. The effect of variety and refrigerated storage in applesauce yield and color was further assessed and reported.

Materials and Methods

Apples

Apples (*Malus domestica* Borkh.) that included Ben Davis, Cortland, Crispin, Empire, Golden Delicious, Idared, Jonagold, McIntosh, Rhode Island Greening and Rome Beauty were harvested between September and November of 2009 from apple farms located in New York State and delivered to the processing pilot plant at Cornell University. Apples were kept under cold storage (CS – 1 °C and 95 % relative humidity) until processing day, executed monthly (starting in November), for up to 8 months.

Apple Maturity Indicators and Applesauce Processing

Prior to processing, apples were tested for firmness using a hand-held penetrometer model FT 327 (Wagner Instruments, Greenwich, CT). Sauce making followed a hot-break method as suggested by Wiley and Binkley (1989) with slight modifications, summarized in Figure 2.1. A sample of filtered exudate from comminuted apple slices was tested for pH using a bench-top Thermo Scientific pH meter model Orion 3-Star (Cellomics, Pittsburgh, PA); titratable acidity (TA) – through titration with NaOH 0.1 N and recorded as % malic acid; soluble solids – according to AOAC (2000) utilizing a bench-top refractometer model Leica Auto Abbe (Leica Inc., Buffalo, NY); and apple slices were assayed for starch content (%) by Dairy One Forage Laboratory (Dairy One Cooperative Inc., Ithaca, NY). Total sauce yield (%) was recorded as the weight of steamed apple slices over the total weight of apples.

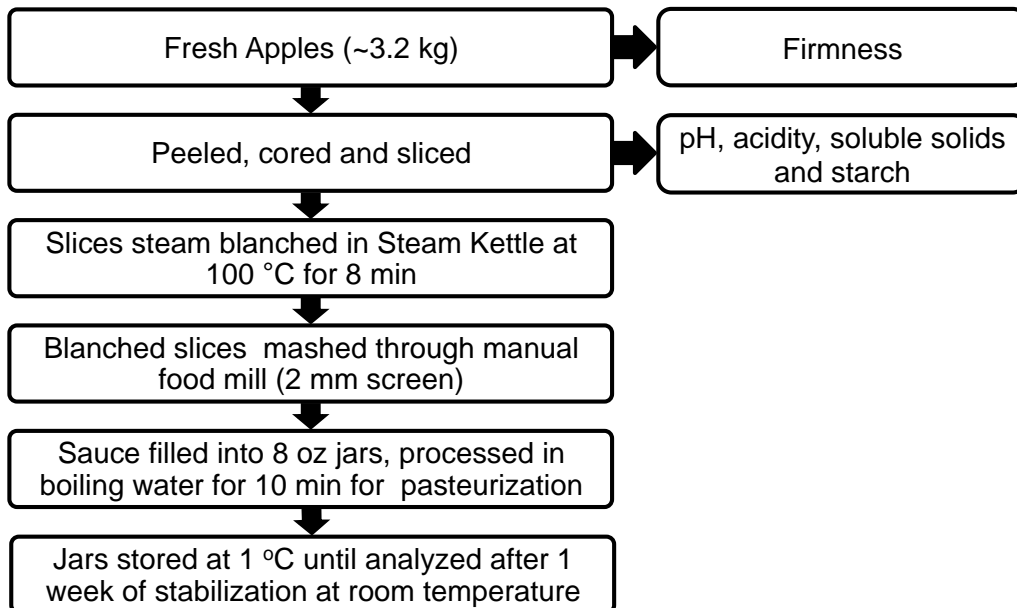


Figure 2.1 – Hot-break applesauce processing diagram.

Final Product Analysis

Applesauce

Applesauce rheological properties yield stress and consistency index were determined using a vane spindle model V-74 in a Brookfield DV-III Ultra programmable rheometer at constant temperature (25 °C) with software packages EZ-Yield and RheoCalc, respectively (equipment and programs from Brookfield Engineering Laboratories, INC. Middleboro, MA). The spindle was introduced in undisturbed applesauce samples, which were torqued slowly recording shear-stress overtime until a yield stress was attained by static vane-based method. Consistency Index was calculated using the power law model; by subjecting samples to 0.5 s⁻¹ increments of shear-rate from 0.5 to 3.0 s⁻¹ upward and backward with 1 min hold at each shear-rate point prior to recording shear-stress and viscosity of samples every 1-min, during a total time of 11 min. USDA consistency was measured according to the Grading Manual for Canned Applesauce (USDA, 2009) and qualitative consistency grading was assigned. The volume-based particle size distribution (PSD) Mean particle size (MPS) and particle size distribution span (PSDS) were assessed using a Malvern laser diffraction unit model Mastersizer 2000 (Malvern Instruments Inc., Westborough, MA). MPS was calculated as the volume-based mean particle diameter ($\sum_i n_i d_i^4 / \sum_i n_i d_i^3$ where n_i is the number of particles of diameter d_i) and PSDS was calculated as the width of the volume-based particle size distribution $((d_{90th\ percentile} - d_{10th\ percentile}) / d_{50th\ percentile})$. The Hunter L, a', and b' color components of applesauce color were measured in a 2 cm glass cuvette by a HunterLab Ultra Scan XE spectrophotometer (Hunter Associates Laboratory, Inc., Reston, VA) set on reflectance mode. Applesauce moisture was obtained according to

AOAC (2000). Applesauce pH was obtained as previously described for apple slices. Applesauce samples were centrifuged at 17000 rpm for 30 min and the supernatant (applesauce serum) was collected and stored at -10 °C until further analysis. Determination of alcohol insoluble solids (AIR) in applesauce, total soluble pectin (TSP) and pectin degree of methoxylation (PDM) followed the methods used by Fraeye and others (2009) with slight modifications. Isolation of AIR in applesauce was carried out according to Mcfeeters & Armstrong (1984) by homogenization of 1 mass unit of applesauce (50 g) in 5 units of 95% ethanol followed by filtration; subsequent ethanol and acetone wash (2.5 units each) and drying at 40 °C until constant weight, reported as % AIR in applesauce. AIR was ground and pulverized for extraction of water- and chelator-soluble pectin fractions - WSP and CSP, respectively. The WSP fraction was obtained by adding 0.25 g of AIR to boiling deionized water; boiling of the mixture for additional 5 min on a heated stirring plate; followed by cooling under running tap water, filtration and final volume adjustment to 50 mL based on the procedure by Sila and others (2006). The CSP fraction was obtained by suspending the residue from WSP filtration with 0.05 M ethylenediaminetetraacetic acid (EDTA) in 0.1 M potassium acetate pH 6.5; which was shaken for 6 h at room temperature followed by filtration and final volume adjustment to 50mL using the same solution according to Chin and others (1999). Each fraction was analyzed for Galacturonic acid (GalA) and methanol for determining pectin content (as galacturonic acid equivalent) and pectin degree of methoxylation (as the ratio of the molar amount of methanol esters to the molar amount of galacturonic acid residues). The galacturonic acid content in pectin fractions was determined by hydrolysis in H₂SO₄/ tetraborate solution (0.0125 M solution of sodium

tetraborate in concentrated sulfuric acid) as described by Ahmed and Labavitch (1977) with subsequent colorimetric determination according to Blumenkrantz and Asboe-Hansen (1973) by using a Barnstead Turner SP830 Spectrophotometer (Barnstead International, Dubuque, IA). The methanol concentration was determined by alkaline hydrolysis of 1 volume of sample in 2 volumes of 0.5 M NaOH and subsequent incubation at room temperature for 1 hour followed by neutralization with 1 volume of 1 M HCl according to Anthon and Barrett (2008). The amount of methanol was determined using alcohol oxidase and Purpald as described by Anthon and Barrett (2004). WSP and CSP were proportionally combined as fractions of applesauce AIR in order to obtain values for total soluble pectin (TSP) and overall pectin degree of methoxylation (PDM) of sauces.

Applesauce Serum

Titrateable acidity was obtained as previously described for apples. Serum samples were assessed for capillary viscosity using a set of Cannon glass capillary viscometers (model Cannon-Fenske routine, Cannon instrument company, State College, PA) calibrated with Brookfield viscosity standards (Brookfield Engineering Laboratories, INC. Middleboro, MA). For determination of AIR content in serum, 1 mass unit of sample was submitted to precipitation with 5 units of 95 % ethanol. The precipitate was filtered through Whatman filter paper 55 mm (Whatman, Piscataway, NJ), dried in oven at 70 °C until stable, weighed and recorded as % AIR. Calcium concentration was determined using Calcium-Arsenazo quantification kit (BEN Biochemical Enterprise, Milano, Italy).

Statistical Analysis

Two batches of apples were processed into sauce generating two replicates each, resulting in a total of 4 samples for each experimental point. Measurement for all experimental units was conducted in duplicate and results were expressed as means and standard deviations. Data was analyzed by ANOVA and significant differences among means adopting a 95 % confidence interval ($p \leq 0.05$) were determined by Tukey's test using JMP® 9.0 statistical software (SAS institute Inc., Cary, NC).

Results and Discussion

Hot-Break Applesauce Consistency

Most varieties produced Grade A applesauce with product flow < 6.5 cm and free-liquid ≤ 1.0 cm. Fruit variety, cold storage and their interaction significantly affected applesauce consistency ($p \leq 0.0001$). Overall, varieties could be divided in two groups according to sauce flow: thicker sauce varieties showing lower sauce flow averaging 2.1-2.5 cm (Ben Davis = Golden Delicious = Rhode Island Greening = Rome Beauty = Crispin) and thinner sauce varieties showing higher sauce flow averaging 3.0-3.6 cm (Idared \leq Cortland = Empire = Jonagold \leq McIntosh) – Figure 2.2 (a) and (b), respectively. Free-liquid was not observed for any of the thicker sauce varieties but was present for Empire, Idared and Jonagold with progressing storage ranging 0.2-1.2 cm. This might indicate that thicker sauce varieties can be used throughout the storage life of the fruit for achievement of products of optimal consistency while thinner sauce varieties

should be preferably used in the beginning of the processing year and/or in lower proportions of fruit blend upon progress of storage.

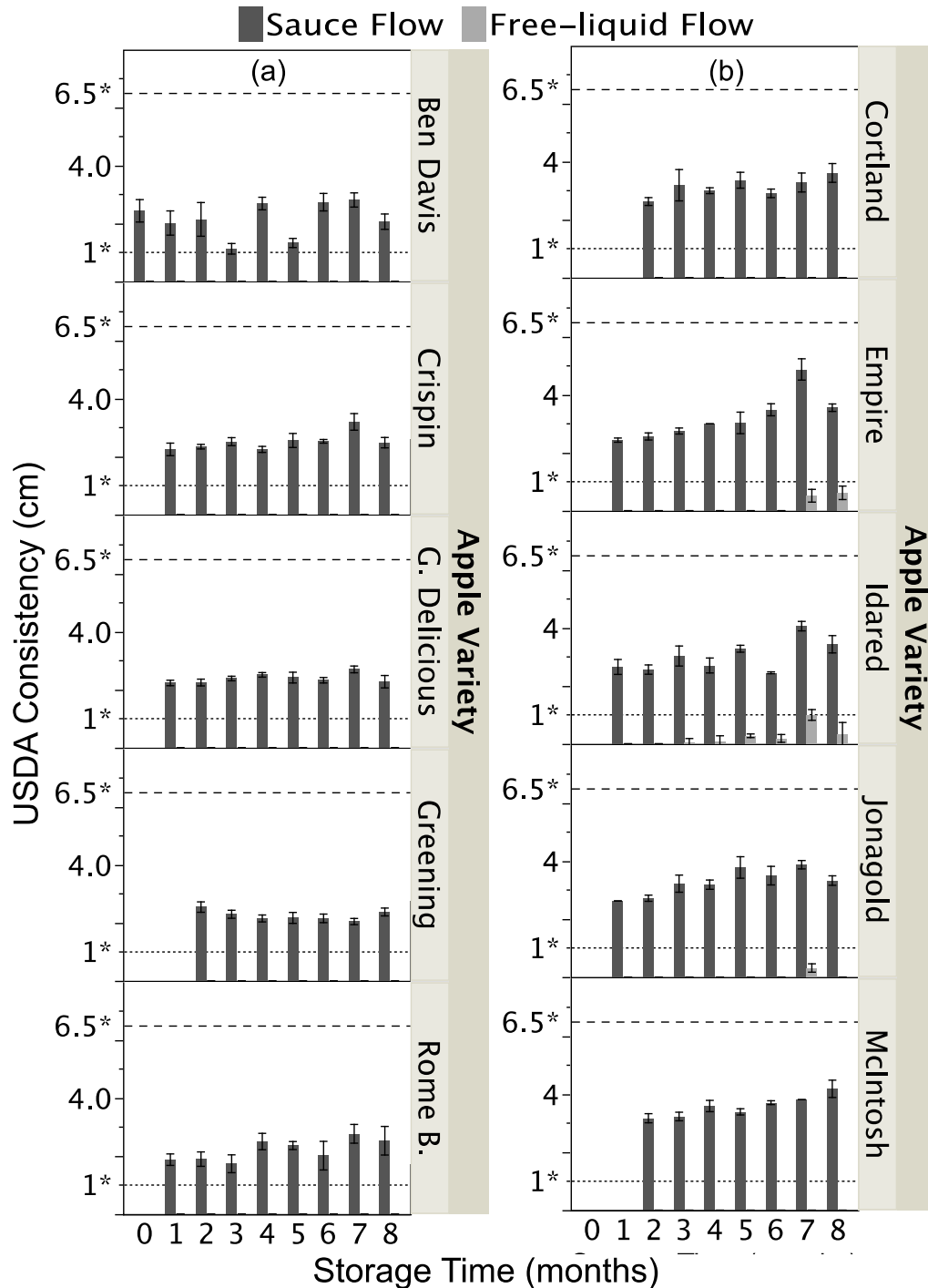


Figure 2.2 – USDA consistency (sauce and free-liquid flow) of applesauce made monthly from apples stored at 1 °C and 95% relative humidity over 8 months of post-harvest storage time – (a) thicker sauce varieties; (b) thinner sauce varieties. *6.5 and 1 cm are tracking parameters for sauce consistency grading.

Result ranges for sauce flow are in agreement with findings by Usiak and others (1995), who studied applesauce made by hot-break (free-liquid was not observed or not reported). These results differ greatly from commercial applesauce samples, with sauce flow observed to be close to 6.5 cm. According to La Belle (1981), commercially, applesauce consistency is adjusted by water incorporated as condensate (about 15 % w/w) or by introduction as an ingredient along with sugar to provide proper flow characteristics and mouth-feel. The present study did not seek these adjustments in order to better observe varietal and storage time effects to product consistency.

Apple Ripening Indicators & Yield in Applesauce

Optimum ripening stage of fruits for applesauce processing is not completely understood. Maturity tests are performed in apples on the field to identify harvest dates for optimum storage potential (Wiley and Binkley, 1989). At processing plants, ripening tests are carried out for assessing fruit quality during storage (Johnston and others, 2002).

Firmness tests indicate significant fruit tissue softening ($p \leq 0.0001$) with extended storage for most varieties and that thicker sauce apples are overall firmer than thinner sauce varieties at the beginning of the storage period – Figure 2.3 (a) and (b) respectively. According to Voragen and others (2003), the ripening process of most fruits is characterized by a decrease in fruit firmness linked to the activity of cell-wall degrading enzymes.

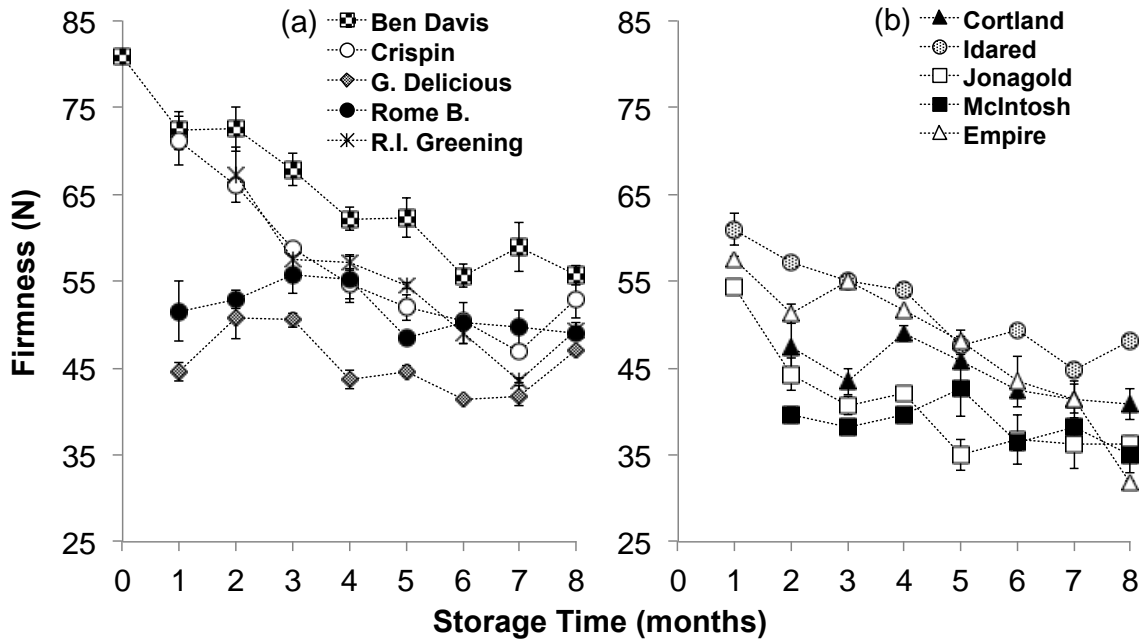


Figure 2.3 – Firmness of apples stored at 1 °C and 95% relative humidity over 8 months of post-harvest storage time – (a) thicker sauce varieties; (b) thinner sauce varieties.

Small amounts of starch can be left in fruits upon harvesting, or none at all (Belitz 2009). Starch levels in apples were insignificant ($\leq 0.2\%$) for all varieties. In our study, applesauce started being processed and analyzed in November, when most of the apple harvest (Sep-Nov) was over. Only the variety Ben Davis was processed freshly harvested, while Cortland, McIntosh and R.I. Greening (harvested in September) and remaining varieties (harvested in October) were kept under cold storage for two and one months, respectively, prior to first experimental point assessment.

Acidity levels decreased as pH increased over storage time for all varieties with similar trends and were not significantly different for both groups ranging 0.72-0.12% and 3.13-4.4, respectively. Applesauce soluble solids was dependent on apple variety being significantly higher for thicker sauce varieties ranging 8.8-13 °Brix. Result ranges were in agreement to what has been reported in the literature (Smock and Neubert, 1950; La Belle, 1981; Massey Jr., 1989).

Significant differences ($p < 0.0001$) were also found for total yield in applesauce (final product output) of different apple varieties, with thicker sauce varieties showing overall higher product yield than thinner sauce group ranging from 42.3 ± 3.5 to $54.9 \pm 3.1\%$ (Empire \leq McIntosh \leq Jonagold G. = Delicious \leq Cortland = R.I.Greening \leq Crispin = Ben Davis = Idared = Rome B.).

Rheological Properties of Hot-Break Applesauce

Yield stress is an important physical property of semi-liquid foods, which denotes the applied stress required to initiate shear flow (Campanella and Pelleg, 1987). As a non-Newtonian fluid, applesauce apparent viscosity can only be described as a function of shear stress, shear rate and temperature (Sahin and Sumnu, 2008). The power law model is commonly used to describe applesauce viscosity (Rao, 2005; Ortega-Rivas, 2012).

Applesauce yield stress was significantly affected by fruit variety, storage time and their interaction ($p \leq 0.001$). Thicker applesauce had higher yield stress compared to thinner – averaging 411 ± 108 vs. 291 ± 96 Pa respectively. Negative correlation was found between sauce USDA consistency (sauce flow) and yield stress (Figure 2.4), indicating applesauce manufacturers could employ the analysis as a direct measurement for assessment of product flow properties facilitating quality control procedures.

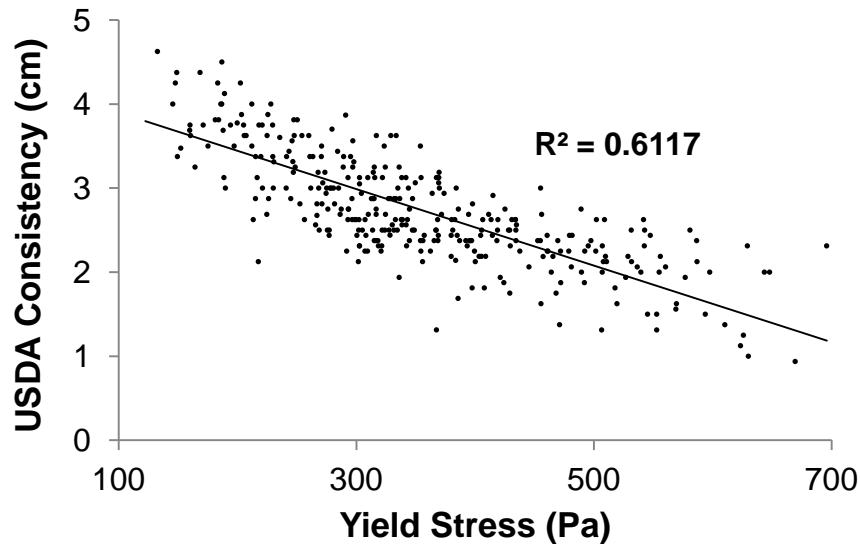


Figure 2.4 – Correlation between USDA consistency (sauce flow) and yield stress of applesauce made monthly from apples stored at 1 °C and 95% relative humidity over 8 months of post-harvest storage time (n =320).

Additionally, the power law model satisfactorily described applesauce rheology with confidence of fit $\geq 77.6\%$ for all samples, all shear-thinning ($n < 1$), in agreement with previous reports from Rao and others (1986), but strong negative correlation with USDA consistency could only be found for Ben Davis ($R^2 = 0.62$). Range of results for yield stress (122 – 695 Pa) and consistency index (53 – 380 Pa.s) was higher than those reported in the literature – 31-87 Pa and 7-50 Pa.s, respectively (Barbosa-Cánovas and Peleg, 1983; Rao and others, 1986; Qiu and Rao, 1988 and 1989; Shijvens and others, 1998). Variations can be attributed to the different processing methods applied in their work to obtain sauce, including reconstitution and soluble solids adjustment; to different rheological assessment methods; and/or to variations inherent to horticultural products in terms of varieties and seasonal changes.

Serum capillary viscosity of sauces significantly decreased with apple storage time ranging 117-3 mPa.s – Figure 2.5 (a) and (b). Values were overall higher for thicker than thinner sauce – averaging 12.3 ± 5.5 vs. 9.4 ± 3.9 mPa.s, respectively after 3 months of

fruit storage, when a plateau was reached for most varieties. Free-liquid flow with progress of storage occurred for varieties with lowest capillary viscosity from thinner sauce group, but no strong correlation was found between the measurement and sauce consistency. This is in agreement with previous studies by Rao and others (1986), who reported that serum viscosity played a minor role in determining the magnitude of apparent viscosity of hot-break applesauce.

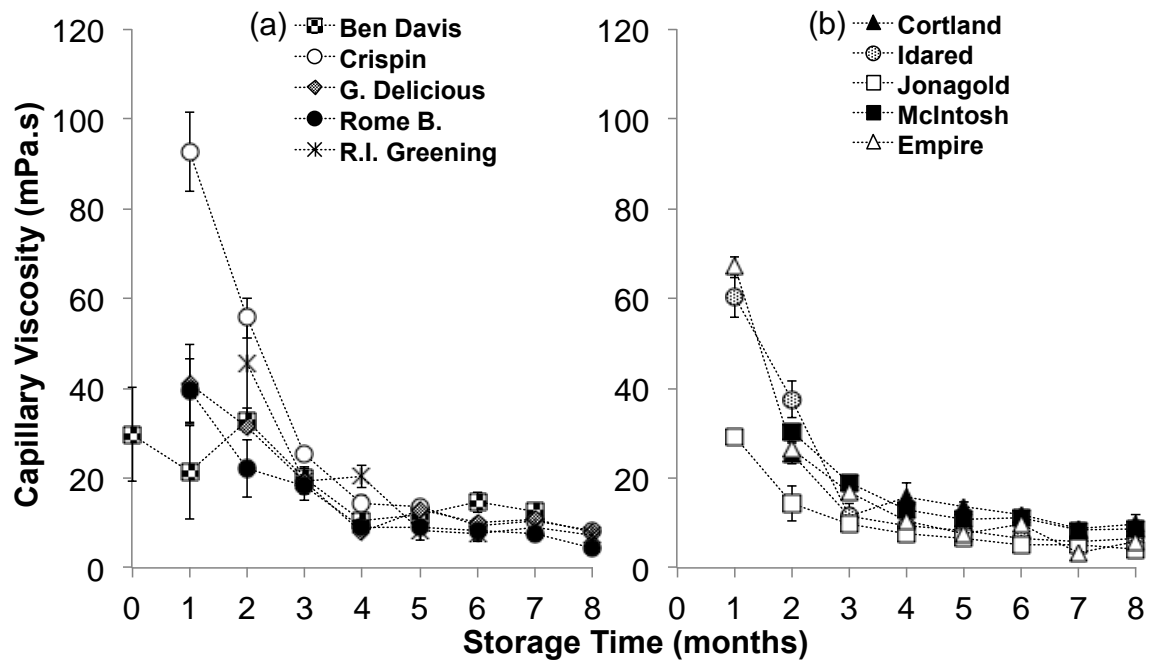


Figure 2.5 – Capillary viscosity of serum collected from applesauce made monthly from apples stored at 1 °C and 95% relative humidity over 8 months of fruit post-harvest storage time – (a) thicker sauce varieties; (b) thinner sauce varieties.

Applesauce Particle Characteristics: Size and Distribution

Figure 2.6 (a) and (b) illustrate the full particle size distribution for thicker and thinner sauce varieties over 4 months of fruit storage (months 2-6 into CS). Mean particle size (MPS) and particle size distribution span (PSDS) significantly affected yield stress and sauce flow of hot-break applesauce. MPS was significantly smaller and PSDS

significantly wider, respectively ($p\text{-value} \leq 0.0001$), for thicker sauce varieties in comparison to thinner sauce ones – ranging 550-772 μm and 1.8-2.2 v.s 670-842 μm and 1.5-1.9, having higher yield stress and lower sauce flow. This is in agreement with reports by Qiu and Rao (1988) on the negative correlation between average particle diameter and applesauce yield stress.

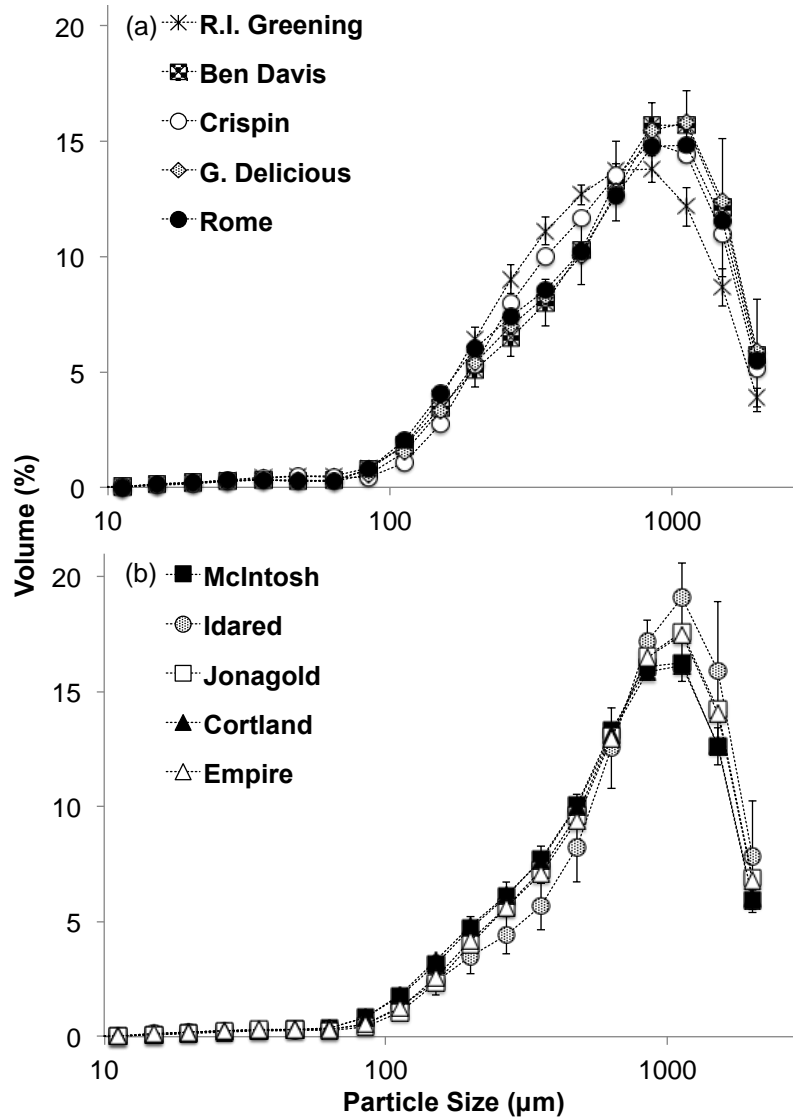


Figure 2.6 – Particle size distribution of applesauce made monthly from apples stored at 1 °C and 95% relative humidity (CS) over 4 months of fruit post-harvest storage time (months 2-6 in CS) – (a) thicker sauce varieties; (b) thinner sauce varieties.

Hot-Break Applesauce Chemical Composition

The fully ripe apple is a complex food matrix composed of about 84% moisture and 16% solids, among which: carbohydrates, nitrogen compounds, fatty materials, vitamins, minerals, astringents, color compounds, enzymes, organic acids and volatiles (Watada and Abbott, 1985). For the study of applesauce consistency, it is important to evaluate moisture, starch, calcium and pectic substances.

Moisture assessment is of interest due to the role of pulp content in rheological properties of sauces (Beresovsky and others, 1995), with special regards to sauce yield stress and therefore consistency. Statistical analysis of moisture points out to a slight, yet significant difference based on apple varieties: higher for thin sauce varieties – $87.6 \pm 0.9\%$ – and lower for thick sauce varieties – $86.4 \pm 0.8\%$. It was a significant factor for rheological properties: higher moisture varieties had higher sauce flow and lower consistency index and yield stress. This is in agreement with reports from Metzner (1985), Tanglerpaibul and Rao (1987) and Qiu and Rao (1988), on the positive correlation between the magnitude of pulp content and sauce yield stress.

Investigation of starch content in applesauce is important given the thickening properties of the starch molecule (Mason, 2009). Starch levels in applesauce serum were insignificant ($\leq 0.2\%$), as a result of its absence in raw fruit.

Calcium is present in apples associated with the cell-wall middle lamella. Apples, apple juice and pulp are reported to have between 2-13 mg/100 g or 20-130 mg/L (ppm) on a fresh weight basis (Perring, 1974; Nour and others, 2010). Calcium ranged 21-26 ppm across all varieties and did not significantly impact applesauce consistency, suggesting sufficient levels are available in applesauce to interact with pectin as

suggested by Pilgrim and others (1991), who reported that the calcium requirement for jellification of low-methoxyl pectins in the presence of calcium averages 20 mg/g.

The residue after alcohol wash – alcohol insoluble residue (AIR) – consists of polysaccharides such as pectic substances together with a small amount of proteins (Ladaniya, 2008). Their assessment is of interest due to their thickening properties of food systems and that of sedimentation prevention, or liquid separation (Stephen and Williams, 2006). AIR was measured in both applesauce (5 selected varieties) and its serum (all 10 varieties). AIR in applesauce serum significantly decreased with progress of storage ranging 3.73-0.13%. It was significantly higher ($p \leq 0.05$) for thicker than thinner applesauce averaging 0.71 ± 0.35 and $0.62 \pm 0.26\%$ respectively, after 3 months of fruit storage, when a plateau was reached, significantly affecting serum capillary viscosity ($p \leq 0.0001$). It was also a significant factor for sauce flow and yield stress, being higher in sauce having lower sauce flow and higher yield stress.

Table 2.1 shows further analysis of AIR, total soluble pectin (TSP) and pectin degree of methoxylation (PDM) in applesauce made from 5 varieties representative of trends observed in sauce consistency (thick and thin sauce showing or not free-liquid flow with progress of storage), over 4 months of storage (2-6 months in CS).

Table 2.1. Changes in chemical parameters of applesauce made from apples stored at 1 °C and 95% relative humidity (CS) over 4 months of fruit storage (2-6 months in CS): alcohol insoluble residue (AIR), total soluble pectin (TSP) and pectin degree of methoxylation (PDM).

Parameter	Apple Variety	Storage Time (months)		
		2	4	6
AIR (%)	Ben Davis	2.48 ± 0.23 ^a	3.39 ± 0.11 ^a	3.28 ± 0.14 ^a
	Idared	2.62 ± 0.21 ^a	1.82 ± 0.03 ^b	1.79 ± 0.04 ^b
	Jonagold	2.74 ± 0.19 ^b	4.01 ± 0.31 ^a	3.24 ± 0.39 ^b
	McIntosh	2.26 ± 0.08 ^a	2.51 ± 0.08 ^b	2.56 ± 0.03 ^{ab}
	R.I. Greening	2.98 ± 0.02 ^b	2.90 ± 0.15 ^b	3.73 ± 0.27 ^a
TSP (%)	Ben Davis	0.24 ± 0.01 ^b	0.31 ± 0.01 ^a	0.21 ± 0.04 ^c
	Idared	0.49 ± 0.11 ^a	0.18 ± 0.01 ^a	0.22 ± 0.03 ^a
	Jonagold	0.21 ± 0.01 ^a	0.19 ± 0.03 ^{ab}	0.15 ± 0.03 ^b
	McIntosh	0.72 ± 0.02 ^a	0.62 ± 0.01 ^b	0.69 ± 0.06 ^{ab}
	R.I. Greening	0.37 ± 0.03 ^{ab}	0.34 ± 0.04 ^b	0.40 ± 0.02 ^a
PDM (%)	Ben Davis	55.2 ± 18.5 ^a	62.0 ± 6.7 ^a	74.5 ± 10.0 ^a
	Idared	43.9 ± 4.9 ^b	43.8 ± 1.2 ^a	40.9 ± 0.94 ^a
	Jonagold	61.8 ± 2.1 ^b	71.1 ± 5.6 ^a	73.0 ± 1.0 ^a
	McIntosh	38.3 ± 0.1 ^a	41.1 ± 0.3 ^c	40.8 ± 0.4 ^b
	R.I. Greening	68.8 ± 13.4 ^a	56.0 ± 0.2 ^a	57.5 ± 0.5 ^a

AIR, TSP and PDM were overall dependent on apple variety and storage time but only TSP significantly affected rheological properties of hot-break applesauce, being higher in sauce showing higher yield stress. In addition, higher sauce flow and free-liquid in thin applesauce varieties seem to be related to low levels of TSP or its degradation with progress of storage as observed for the varieties Idared and Jonagold. Range of results observed for TSP and PDM were overall consistent with previous reports in the literature for apples and applesauce with progress of storage ranging 0.17 – 0.74% and 47 – 88% respectively (Toldby and Wiley, 1962; McClendon and others, 1959; De Vries, 1981; Klein and others, 1995; Johnston and others, 2002; Lo Scalzo and others, 2005; Vanoli and others, 2009; Le Bourvellec and others, 2011; Rascón-Chu and others, 2009).

Applesauce Color

Because flavor differences in applesauce are minimized by the addition of other ingredients such as sweeteners, manufacturers put more emphasis on color and consistency as factors for quality control of original and all-natural products (no flavor or color added).

Figure 2.7 summarizes our findings on the impact of fruit variety on applesauce color carried out with five different apple varieties over 4 months of fruit storage (2-6 months in CS): Jonagold stood out as the lightest sauce (higher L values) while other varieties studied were similar in lightness with Greening being the darkest among them. Overall, there were no changes in this parameter over storage, except for Ben Davis, which became lighter with ripening. Ben Davis, Greening and McIntosh scored the lowest a-values respectively (all negative) indicating green shades, while Idared and Jonagold scored the highest positive a values, associated with red shades, probably due to some leaching of peel pieces. All b-values measured were positive, highlighting the predominant yellow color of sauces but differences in intensity were found: Ben Davis had the highest values and Idared the lowest with other varieties statistically identical in the middle.

Our results did not compare in magnitude with those reported by Luh and Kamber (1963) on color changes of Gravenstein applesauce. Other than the variety, the processing method employed to obtain applesauce in their study was different: water and sucrose were part of the product formulation.

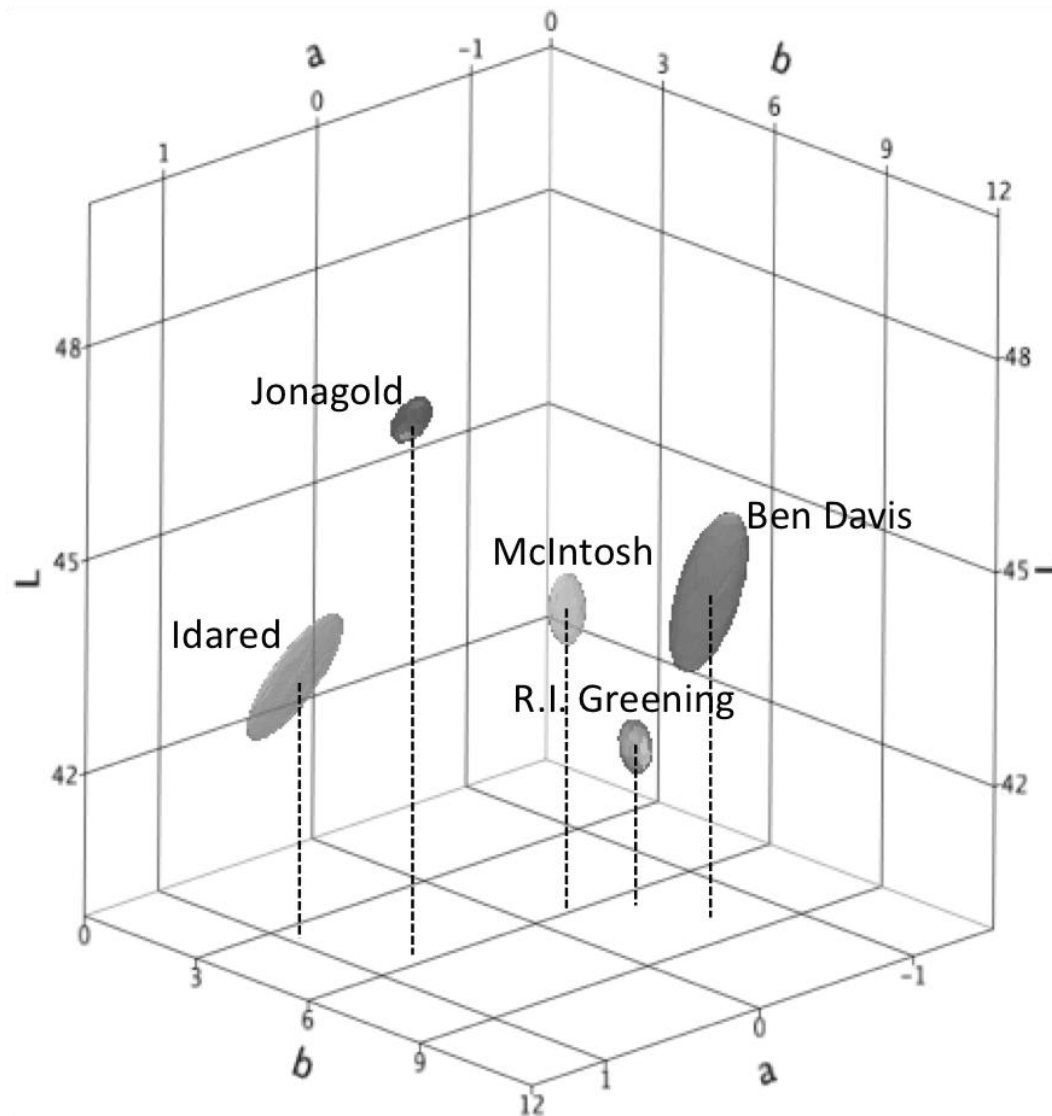


Figure 2.7 – Orthographic 3D Scatterplot of L, a and b color parameters values of applesauce made monthly from apples stored at 1 °C and 95% relative humidity (CS) over 4 months of fruit post-harvest storage time (months 2-6 in CS).

Conclusions

The consistency of hot-break applesauce was significantly affected by apple variety and refrigerated storage time (1 °C and 95 % relative humidity). Applesauce yield stress was correlated with sauce flow, indicating manufacturers can use it as a direct measurement for control of sauce consistency. MPS and PSDS significantly affected

rheological properties of sauce, making it a potential tracking parameter for quality control purposes of consistency of hot-break applesauce. In addition, higher sauce flow and liquid separation with progress of storage was observed in sauce having low AIR in applesauce serum and low TSP pectin levels in sauce, measurements related to the pectin content of products. AIR assessment in applesauce serum involves few equipment requirements and could be additionally employed by manufacturers.

References

- Ahmed A, Labavitch, J. 1977. A simplified method for accurate determination of cell wall uronide content. *J Food Biochem* 1:361–365.
- Anthon, G. E., & Barrett, D. M. 2004. Comparison of three colorimetric reagents for the determination of methanol with alcohol oxidase. Application to the assay of pectin methylesterase. *J Agric Food Chem* 52: 3749–3753.
- Anthon GE, Barrett DM. 2008. Combined enzymatic and colorimetric method for determining the uronic acid and methylester content of pectin: Application to tomato products. *Food Chem* 110(1):239-47.
- AOAC International. 2000. *Official Methods of Analysis of AOAC International*. 17th ed. Arlington: AOAC International. 2200 p.
- Barbosa-Cánovas GV, Peleg M. 1983. Flow parameters of selected commercial semi-liquid food products. *J Texture Stud* 14(3):213-34.
- Belitz HD, Grosch W, Schieberle P. 2009. *Food Chemistry*. 4th ed. New York: Springer. 1070 p.

- Beresovsky N, Kopelman IJ, Mizrahi S. 1995. The role of pulp interparticle interaction in determining tomato juice viscosity. *J Food Process Preserv* 19(2):133-46.
- Blumenkrantz, N., & Asboe-Hansen, G. 1973. New method for quantitative determination of uronic acids. *Anal Biochem* 54: 484–489.
- Brownleader M, Jackson P, Mobasher A, Pantelides A, Sumar S, Trevan M, Dey P. 1999. Molecular aspects of cell wall modifications during fruit ripening. *Crit Rev Food Sci Nutr* 39(2):149-64.
- Burg SP. 2004. *Postharvest Physiology and Hypobaric Storage of Fresh Produce*. Cambridge: CABI Publishing. 654 p.
- Campanella OH, Peleg M. 1987. Determination of the yield stress of semi-liquid foods from squeezing flow data. *J Food Sci* 52(1):214-5.
- Chin, L. H., Ali, Z. M., & Lazan, H. 1999. Cell wall modifications, degrading enzymes and softening of carambola fruit during ripening. *J Exp Bot* 50(335): 767–775.
- De Vries JA, Voragen AGJ, Rombouts FM, Pilnik W. 1981. Extraction and purification of pectins from Alcohol Insoluble Solids from ripe and unripe apples. *Carbohydr.Polym* 1(2):117-27.
- FDA: 21CFR145.110 – Canned Applesauce [Internet]. Silver Spring, MD: U.S. Food and Drug Administration [Accessed 2012 Sep 17]. Available from: <http://www.accessdata.fda.gov/scripts/cdrh/cfdocs/cfcfr/CFRSearch.cfm?fr=145.110>.
- Fraeye I, Doungra E, Duvetter T, Moldenaers P, Van Loey A, Hendrickx M. 2009. Influence of intrinsic and extrinsic factors on rheology of pectin–calcium gels. *Food Hydrocolloid* 23(8):2069-77.

- Johnston J, Hewett E, Hertog M. 2002. Postharvest softening of apple (*Malus domestica*) fruit: A review. N Z J Crop Hortic Sci 30(3):145-60.
- Kader AA. 1986. Biochemical and physiological basis for effects of controlled and modified atmospheres on fruits and vegetables. Food Technol 40(5):99-100, 102-104.
- Klein JD, Hanzon J, Irwin PL, Shalom NB, Luria S. 1995. Pectin esterase activity and pectin methyl esterification in heated golden delicious apples. Phytochemistry 39(3):491-494.
- La Belle RL. 1981. Apple quality characteristics as related to various processed products. In: R. Teranishi and H. Barrera-Benitez. Quality of selected fruits and vegetables of North America. ACS Symposium Series. Washington: American Chemical Society. p. 61–76.
- Ladaniya MS. 2008. Citrus fruit: biology, technology and evaluation. San Diego: Academic Press. 558p.
- Le Bourvellec C, Bouzerzour K, Ginies C, Regis S, Plé Y, Renard CMGC. 2011. Phenolic and polysaccharidic composition of applesauce is close to that of apple flesh. Journal of Food Composition and Analysis 24(4–5):537-47.
- Lee YS, Salunkhe DK, Do JY, Olson LE. 1966. Physiological and Biochemical Factors Influencing Quality of Canned Applesauce. Proceedings of the American Society for Horticultural Science. June. 88:116.
- Lo Scalzo R, Forni E, Lupi D, Giudetti G, Testoni A. 2005. Changes of pectic composition of ‘annurca’ apple fruit after storage. Food Chem 93(3):521-530.

- Louis M, Massey JR. 1989. Harvesting, Storing and Handling Processing Apples. In: Downing DL. Processed apple products. New York: Van Nostrand Reinhold. P 215-238.
- Luh BS, Kamber PJ. 1963. Chemical and color changes in canned apple sauce. Food Technol 17:105-106.
- Mason WR. 2009. Starch use in foods. In: BeMiller J, Whistler R. Starch. 3rd ed. San Diego: Academic Press. p 745-95.
- Massey Jr. LM. 1989. Harvesting, storing and handling processing apples. In: Downing DL. Processed apple products. New York: Van Nostrand Reinhold. P 31-51.
- McClendon JH, Woodmansee CW, Somers GF. 1959. On the occurrence of free-galacturonic acid in apples and tomatoes. Plant Physiol 34(4):389-91.
- Mcfeeters, R. F., & Armstrong, S. A. (1984). Measurement of pectin methylation in plant-cell walls. Anal Bio 139(1): 212–217.
- McClendon JH, Woodmansee CW, Somers GF. 1959. On the occurrence of free-galacturonic acid in apples and tomatoes. Plant Physiol 34(4):389-91.
- Metzner AB. 1985. Rheology of suspensions in polymeric liquids. J Rheol 29(6): 739-775.
- Mohr WP. 1973. Applesauce grain. J Texture Stud 4(2):263-8.
- Mohr WP. 1989. Influence of cultivar, fruit maturity, and fruit anatomy on apple sauce particle size and texture. Int J Food Sci Technol 24(4): 403-413.
- New York Apple Association: New York apples fast facts [Internet]. Victor, NY: New York Apple Association [Accessed 2011 Jun 13]. Available from: <http://www.nyapplecountry.com/fastfacts.htm>.

- Nogueira JN, McLellan MR, Anantheswaran RC. 1985. Effect of Fruit Firmness and Processing Parameters on the Particle Size Distribution in Applesauce of Two Cultivars. *J Food Sci* 50(3):744-6.
- Nour V, Trandafir I, Ionica ME. 2010. Compositional characteristics of fruits of several apple (*Malus domestica* Borkh.) cultivars. *Not Bot Hort Agrobot Cluj* 38 (3): 228-233.
- Ortega-Rivas E. 2012. Non-thermal food engineering operations. New York: Springer. 375 p.
- Perring MA. 1974. The chemical composition of apples. XI. An extraction technique suitable for the rapid determination of calcium, but not potassium and magnesium, in the fruit. *J Sci Food Agric* 25(3): 237–245.
- Pilgrim GW, Walter RH, Oakenfull DG. 1991. Jams, jellies and preserves. In: Walter RH. *The Chemistry and Technology of Pectin*. San Diego: Academic Press. p 23-50.
- Qiu C, Rao MA. 1988. Role of pulp content and particle size in yield stress of apple sauce. *J Food Sci* 53(4):1165-1170.
- Rao MA. 2005. Rheological properties of fluid foods. In: Rao MA, Rizvi SSH, Datta AK. *Engineering properties of foods*. New York: CRC Press. p 41-98.
- Rao MA, Cooley HJ, Nogueira JN, McLellan MR. 1986. Rheology of apple sauce: effect of apple cultivar, firmness, and processing parameters. *J Food Sci* 51(1):176-179.
- Rascón-Chu A, Martínez-López AL, Carvajal-Millán E, Ponce de León-Renova NE, Márquez-Escalante JA, Romo-Chacón A. 2009. Pectin from low quality ‘golden delicious’ apples: composition and gelling capability. *Food Chem* 116(1):101-103.

- Sila, D. N., Dounghla, E., Smout, C., Van Loey, A., & Hendrickx, M. (2006). Pectin fraction interconversions: Insight into understanding texture evolution of thermally processed carrots. *Journal of Agricultural and Food Chemistry*, 54(22), 8471–8479.
- Schijvens EPHM, Van Vliet T, Van Dijk C. 1998. Effect of Processing Conditions on the composition and rheological properties of applesauce. *J Texture Stud* 29(2):123-143.
- Smock RM, Neubert AM. 1950. Apples and apple products. New York: Interscience Publishers. P 486.
- Stephen AM, Glyn OP, Williams PA. 2006. Food polysaccharides and their applications. New York: CRC Press. 752 p.
- Tanglertpaibul T, Rao MA. 1987. Flow properties of tomato concentrates: effect of serum viscosity and pulp content. *J Food Sci* 52(2): 318–321.
- Toldby V, Willey R. 1962. Liquid-solids separation, a problem in processed applesauce. *J Am Soc Hortic Sci* 81:78-90.
- USDA: U.S. Apple Statistics [Internet]. National Agricultural Statistics Service, Cold Storage Annual Summary, various issues. Washington, D.C.: United States Department of Agriculture [Accessed 2012 Mar 31]. Available from: <http://usda.mannlib.cornell.edu/MannUsda/viewDocumentInfo.do?documentID=1825>.
- USDA: Grading Manual for Canned Applesauce [Internet]. Washington, D.C.: United States Department of Agriculture [Accessed 2009 Sep 19]. Available from: <http://www.usda.gov>.

- Usiak AMG, Bourne MC, Rao MA. 1995. Blanch temperature/time effects on rheological properties of applesauce. *J.Food Sci.* 60(6):1289-1291.
- Vanoli M, Zerbini PE, Spinelli L, Torricelli A, Rizzolo A. 2009. Polyuronide content and correlation to optical properties measured by time-resolved reflectance spectroscopy in 'Jonagored' apples stored in normal and controlled atmosphere. *Food Chem.* 115(4):1450-7.
- Voragen A, Voragen F, Schols H, Visser R. 2003. Advances in pectin and pectinase research. Dordrecht: Kluwer Academic Publishers. 514 p.
- Way RD, McLellan MR. 1989. Apple cultivars for processing. In: Downing DL. *Processed Apple Products*. New York: Van Nostrand Reinhold. p 1-29.
- Watkins, C.B. 2003. Principles and practices of postharvest handling and stress. In: *Apples: Crop Physiology, Production and Uses*. Feree, D. and I.J. Warrington (eds) Chapt. 23, CAB Pub. pp. 585-614.
- Wiley RC, Binkley CR. 1989. Applesauce and other canned apple products. In: Downing DL. *Processed apple products*. New York: Van Nostrand Reinhold. P 215-238.
- New York: Van Nostrand Reinhold. P 215-238.

CHAPTER 3:
EFFECT OF VARIETY AND RIPENESS OF APPLES (*Malus domestica* Borkh.)
ON PHYSICAL AND CHEMICAL PARAMETERS AFFECTING
RHEOLOGICAL PROPERTIES OF COLD-BREAK APPLESAUCE

ABSTRACT: Challenges in achieving products of optimal consistency have been faced by cold-break applesauce manufacturers when processing newly harvested fruit. Five different apple varieties (Crispin, Idared, Jonagold, Rhode Island Greening and Rome Beauty) were used to assess the effect of apple variety and post-harvest fruit ripening on applesauce rheological properties over 2 harvest years (2010 and 2011). Apples were harvested and stored up to 5 months at 1 °C and 95% relative humidity (cold storage – CS). Applesauce was obtained monthly following a cold-break procedure. Apples were evaluated for ripeness (firmness, pH, acidity, soluble solids); and applesauce for rheological properties (USDA consistency, yield stress, consistency index); and physical and chemical parameters – particle size distribution, mean particle size (MPS) and particle size distribution span (PSDS); moisture; calcium; starch; alcohol insoluble residue (AIR); total soluble pectin (TSP) and pectin degree of methoxylation (PDM). Results were analyzed by ANOVA and significant differences among means determined by Tukey’s test ($p \leq 0.05$). Harvest season, apple variety and storage time were significant factors for applesauce rheological properties ($p\text{-value} \leq 0.05$). Yield stress and consistency index improved with progress of storage and were negatively correlated with USDA consistency sauce flow ($R^2 \geq 0.62$). Differences in applesauce rheological properties among varieties, over fruit CS and harvest seasons are explained by differences in MPS and PSDS (ranging 492 – 1061 μm and 1.02 – 2.25 respectively) as well as in starch (0 – 0.78%), AIR (1.59 – 5.69%), TSP (0.13 – 0.53%) and PDM (34.4 – 79.1%). Calcium was not significantly different among varieties and ranged 18 – 37 ppm.

Keywords: applesauce consistency, apple variety, fruit ripening, cold storage.

Practical Application: A more efficient and cost-effective cold-break procedure has been increasingly adopted for applesauce manufacturing, eliminating the energy and time-demanding cooking step of the traditional hot-break processing line. The adoption of the technology has been accompanied by challenges in achieving products of desirable rheological properties specially when processing newly harvested and controlled atmosphere stored fruit. Varietal blending is a common practice utilized by the applesauce industry for the achievement of consistent products throughout the processing year. Information about rheological properties and physical-chemical composition of single-variety applesauce taking post-harvest fruit ripening into consideration can provide valuable information for fruit blend management by cold-break applesauce manufacturers.

Introduction

Applesauce is a typical American product. Traditionally prepared at home, it is nowadays the predominant apple-based canned product in the United States (New York Apple association, 2011) being widely available commercially as a formulated product in family size and single serve units. According to the product identity standards, it is the food prepared from comminuted or chopped apples, which may have added to it ingredients specified by the regulation (FDA, 2012).

In the United States, applesauce manufacturers process apples harvested from August to November (Calvin and Martin, 2011) year round which are kept in cold storage (CS) – 1-4 °C and 95-98% relative humidity (RH) – or controlled atmosphere storage (CA) – 1-3% O₂ and 1-5% CO₂ at 1-4 °C and 95-98% RH – for up to 6 to 12 months or

more, respectively, depending on storage condition (Louis and Massey, 1989; Watkins, 2003; USDA, 2012).

Applesauce quality grading is established through the assessment of 5 attributes: absence of defects, color, flavor, finish and consistency (USDA, 2009). From those, quality control of product consistency – a flow measurement related to the separation of solids and liquid – is particularly important because consumer complaints are often related to excessive free-liquid or thin sauce, which, in addition, can pose challenges at the processing line at the filler step, causing product to overflow the primary package prior to capping and sealing, leading to considerable financial loss for the industry.

Challenges to achieve products of optimal consistency have surfaced after a more efficient and cost-effective cold-break procedure has been adopted by applesauce manufacturers to replace the energy and time-demanding traditional hot-break processing line. The most challenging times are the beginning of the processing year, when newly-harvested fruits are used, and when CA stored fruits start being used latter in the season.

Differences observed in processing performance of freshly harvested fruit vs. fruit stored for longer periods could be related to differences in physical and chemical composition of sauce. According to Varela and others (2007), physical and chemical composition of apples will be dependent on several factors such as variety, climate conditions during fruit growth, stage of maturity at harvest, and post-harvest storage conditions. Furthermore, as climacteric fruits, apples continue to ripen after being harvested, undergoing physical and chemical changes over storage (Burg, 2004; Goulao and Oliveira, 2008) which are known to impact table quality fruit (Perring, 1989;

Konopacka and Plocharski, 2004) and applesauce sensory attributes (Mohr, 1973 and 1989; Lanza and Kramer, 1967; McLellan and Massey Jr., 1984).

Literature on parameters affecting applesauce consistency is scarce, and, similarly to literature on applesauce rheological properties, focus on product obtained by the hot-break procedure. Toldby and Wiley (1962) studied the lyophoresis, or liquid-solid separation in applesauce, and determined that it is related to combined chemical and physical characteristics of sauces such as starch and pectin content and average particle size; Rao and others (1986) reported the effect of apple cultivar, firmness and processing parameters (finisher speed and screens size) on applesauce rheology; Qui and Rao (1988) studied the role of pulp content and particle size on applesauce yield stress; Usiak and others (1995) studied the effect of blanch temperature and time on applesauce rheological properties (USDA consistency and consistency index). Information on rheological properties of sauce obtained by the cold-break procedure and how they relate to product consistency is needed.

In an attempt to even out the impact of fruit composition and natural changes over storage, applesauce manufacturers typically use a blend of different apple varieties for the achievement of consistent products year-round (Wiley and Binkley, 1989). The practice poses an additional challenge to estimate varietal contribution to consistency of final products due to combined effect of varieties and their proportion in a given blend. Post-harvest fruit ripening over storage as well as changes in fruit composition based on different harvest seasons could be additional sources of variation in processing performance of different apple varieties into sauce.

The aim of this study was thus to assess the effect of apple variety and fruit post-harvest ripening under CS on physical and chemical parameters affecting cold-break applesauce rheological properties, with a focus on product consistency. The effect of seasonality and potential effect on sauce rheological properties was addressed by studying applesauce made over 2 harvest years.

Materials and Methods

Apples

Apples (*Malus domestica* Borkh.) that included Crispin, Idared, Jonagold, Rhode Island Greening and Rome Beauty were harvested between September and October of 2010 and 2011 from apple farms located in New York State and delivered to the processing pilot plant at Cornell University where they were kept at 1 °C and 95% relative humidity (RH) – cold storage (CS) – until processing day, carried out monthly (every 28-32 days) for up to 5 months.

Apple Maturity Indicators and Applesauce Processing

Prior to processing, apples were weighed and tested for firmness using a hand-held penetrometer model FT 327 (Wagner Instruments, Greenwich, CT). Sauce making followed a cold-break procedure: apples (~ 15 kg) were fed to a turbo extractor (1.6 mm screen 8 mm gap 1800 rpm; Bertocchi CX5, Bertocchi SLR., Parma, Italy) and 15% water (w/w) was added to the sauce (to simulate water pick-up by direct steam injection) which was then heated in a steam kettle at 96-98 °C for 6 min and hot-filled into 8 oz

glass jars. Jars were inverted for 3 min for cap sterilization, cooled in water bath and stored at 1 °C until analysis. A sample of comminuted apples (turbo extractor output) was collected and pressed through cheesecloth to obtain juice which was tested for pH using a bench-top Thermo Scientific pHmeter model Orion 3-Star (Cellomics, Pittsburgh, PA); titratable acidity (TA) – through titration with NaOH 0.1 N and recorded as % malic acid; and soluble solids – according to AOAC (2000) utilizing a bench-top refractometer model Leica Auto Abbe (Leica Inc., Buffalo, NY).

Applesauce Analysis

Applesauce yield stress and consistency index of samples were determined using a vane spindle model V-73 in a Brookfield DV-III Ultra programmable rheometer at constant temperature (25 °C) with software package RheoCalc (equipment and program from Brookfield Engineering Laboratories, INC. Middleboro, MA). Yield Stress and Consistency Index were calculated using the Casson and power law models, respectively, from shear-stress data obtained by subjecting samples to 0.5 s⁻¹ increments of shear-rate from 0.5 to 3.0 s⁻¹ upward and backward with 1 min hold at each shear-rate prior to data collection every 1-min, during a total time of 11 min. USDA consistency was measured according to the Grading Manual for Canned Applesauce (USDA, 2009) and qualitative consistency grading was assigned. The volume-based particle size distribution (PSD), mean particle size (MPS) and particle size distribution span (PSDS) were assessed using a Malvern laser diffraction unit model Mastersizer 2000 (Malvern Instruments Inc., Westborough, MA). MPS was calculated as the volume-based mean particle diameter ($\sum_i n_i d_i^4 / \sum_i n_i d_i^3$ where n_i is the number of particles of diameter d_i) and PSDS was

calculated as width of the volume-based particle size distribution $((d_{90th\ percentile} - d_{10th\ percentile}) / d_{50th\ percentile})$. Applesauce moisture was obtained according to AOAC (2000). Applesauce pH was obtained as previously described for apple slices. Applesauce samples were centrifuged at 17000 rpm for 30 min and the supernatant (applesauce serum) was collected and stored at -10 °C until further analysis. Alcohol insoluble residue (AIR), total soluble pectin (TSP) and pectin degree of methoxylation (PDM) analysis were carried out as previously described in detail in Chapter 2. Isolation of AIR was carried out according to Mcfeeters & Armstrong (1984) and reported as % AIR in applesauce. AIR was ground and pulverized for extraction of water- and chelator-soluble pectin fractions (WSP and CSP, respectively). The WSP and CSP fractions were obtained following the procedures by Sila and others (2006) and Chin and others (1999), respectively. Each fraction was analyzed for Galacturonic acid (GalA) and methanol for determining pectin content (as GalA equivalent) and pectin degree of methoxylation (as the ratio of the molar amount of methanol esters to the molar amount of galacturonic acid residues). The GalA content in pectin fractions was determined by hydrolysis in H₂SO₄/tetraborate solution (0.0125 M solution of sodium tetraborate in concentrated sulfuric acid) as described by Ahmed and Labavitch (1977) with subsequent colorimetric determination according to Blumenkrantz and Asboe-Hansen (1973) by using a Barnstead Turner SP830 Spectrophotometer (Barnstead International, Dubuque, IA). The methanol concentration was determined by alkaline hydrolysis of 1 volume of sample in 2 volumes of 0.5 M NaOH and subsequent incubation at room temperature for 1 hour followed by neutralization with 1 volume of 1 M HCl according to Anthon and Barrett (2008). The amount of methanol was determined using alcohol oxidase and Purpald as

described by Anthon and Barrett (2004). WSP and CSP were proportionally combined as fractions of applesauce AIR in order to obtain values for TSP and PDM of sauces. Additionally, applesauce samples were centrifuged at 17000 rpm for 30 min and the supernatant (applesauce serum) was collected through filtration also using Whatman filter paper 55 mm. The serum was stored at -10 °C until further analysis. Applesauce serum titratable acidity was obtained as previously described for apples. Calcium concentration in applesauce serum was determined using Calcium-Arsenazo quantification kit (BEN Biochemical Enterprise, Milano, Italy). Starch analysis was performed through iodine-iodide 0.01 N reaction measuring absorbance at 570 nm referring to a standard curve of known starch concentrations in a Turner spectrophotometer model Barnstead SP-830 (Turner Biosystems, Dubuque, IA) reported as % starch (g/100 ml).

Statistical Analysis

Two batches of apples were processed into sauce generating two replicates, resulting in a total of 4 samples for each experimental point. Measurement for all experimental units was conducted in duplicate or triplicate and results were expressed as means and standard deviations. Data was analyzed by ANOVA and significant differences among means adopting a 95 % confidence interval ($p \leq 0.05$) were determined by Tukey's test using JMP® 9.0 statistical software (SAS institute Inc., Cary, NC).

Results and Discussion

Apple Ripening Indicators

Apple firmness results point out to significant fruit tissue softening (p-value \leq 0.05) during the first 2-3 months of cold storage after which time it stabilized ranging from a maximum of 96 N after harvest to a minimum of 36 N after 5 months of fruit storage in CS (Figure 3.1). Firmness and other fruit ripening indicators and trends were in agreement with previous literature (La Belle, 1981; Massey Jr., 1989): soluble solids (9.9 – 16.1 °Brix); titratable acidity (0.969 – 0.152%); pH (3.12 – 3.99). Fruit ripening has long been linked to tissue softening as a result of the action of hydrolytic enzymes on their cell wall carbohydrate components (Brownleader and others, 1999); increase in total sugars due to starch degradation and decline in total titratable acidity followed by an increase in pH (Smock and Neubert, 1950). According to Wiley and Binkley (1989) attempts to use ripening indices to predict the quality of canned applesauce have not been successful due to loss of integrity of the raw apple tissue in sauce processing.

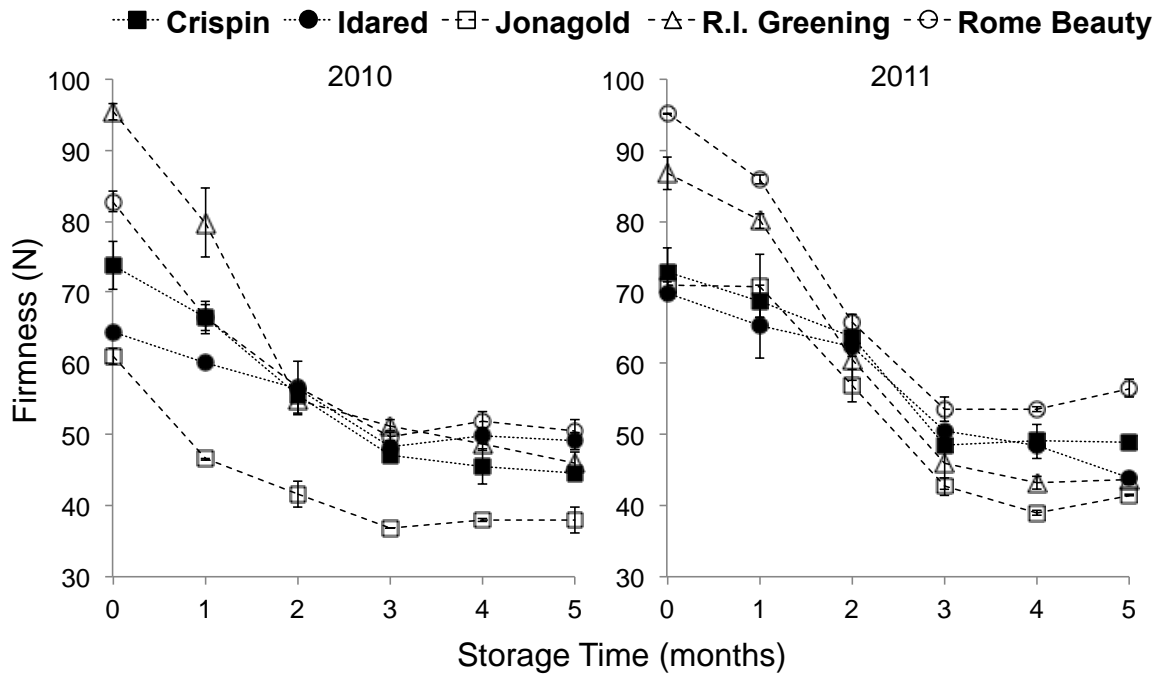


Figure 3.1 – Firmness of apples stored at 1 °C and 95% relative humidity over 5 months of storage, over 2 harvest years.

Applesauce Rheological Properties

USDA Consistency

Applesauce consistency is measured with the USDA aid 105, which is a plastic chart containing concentric circular markings, 0.5 cm apart each for assessment of average sauce and liquid spread of products subjected to flow for 1 min. Free-liquid flow is achieved by subtracting sauce from liquid flow. Grade A consistency sauce flow shall not surpass 6.5 cm and any free-liquid shall not surpass 0.7 cm. Grade B sauce flow shall not surpass 8.5 cm and any free liquid shall not surpass 1 cm. Substandard (SSTD) applesauce fails to meet Grade B requirements (USDA 2009). Consistency results are summarized in Figure 3.2.

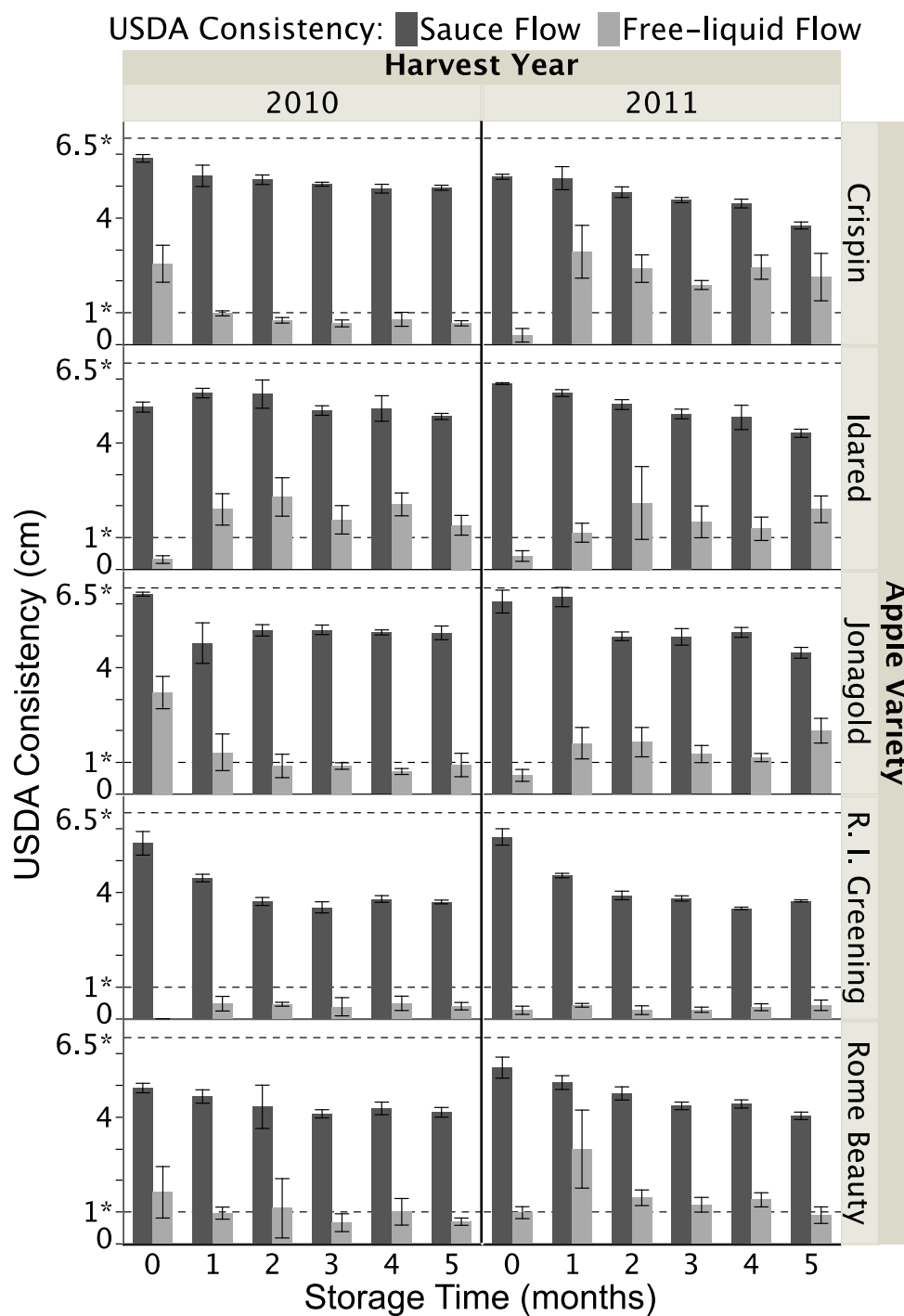


Figure 3.2 – USDA consistency (sauce and free-liquid flow) of applesauce made monthly from apples stored at 1 °C and 95% relative humidity over 5 months of storage, over 2 harvest years. *6.5 and 1 cm are tracking parameters for sauce consistency grading.

Harvest season, apple variety and storage time were significant factors for applesauce consistency, both for sauce and free-liquid flow ($p \leq 0.05$). Sauce flow was \leq

6.5 cm for all varieties over storage improving with 2-3 months of storage time (lower sauce flow readings, indicating thicker sauce) as a general trend while free-liquid quality varied greatly among varieties and with storage time at different harvest years. Rhode Island Greening was the most consistent variety, producing grades A or B applesauce (free liquid ≤ 1.0 cm) and showing similar trends over both years throughout cold storage, suggesting the variety can be used in the sauce blend targeting product consistency optimization. Applesauce made from Rome Beauty also showed similar trends over both harvest years with consistency improvements ($p \leq 0.05$) after 3 months of fruit storage, indicating that the quality of sauce made from those apples can benefit from changes occurring to raw materials under storage. Crispin, Idared and Jonagold were, overall, challenging varieties, yielding SSTD sauce (free-liquid > 1.0 cm) showing storage and harvest year effects. While in 2010 the applesauce made from Crispin and Jonagold apples had improved consistency with progress of storage (free-liquid flow of 2.53 ± 0.58 and 3.19 ± 0.51 cm at the beginning of storage to 0.75 ± 0.08 and 0.88 ± 0.37 cm after 2 months in cold storage respectively and stable thereafter), product consistency decreased in quality in 2011 (free-liquid flow of 0.28 ± 0.26 and 0.58 ± 0.19 cm at the beginning of storage to 2.91 ± 0.83 and 1.59 ± 0.50 cm after 1 month in cold storage and stable or higher thereafter). Idared produced sauce of substandard consistency over the 2010 and 2011 harvest years with progress of storage (free-liquid flow of 0.3 ± 0.12 and 0.41 ± 0.17 cm at the beginning of storage to 1.88 ± 0.49 and 1.14 ± 0.30 cm after 1 month in cold storage and stable thereafter, respectively). Great variability of consistency and presence of free-liquid in the sauce produced by challenging varieties over harvest years

might indicate those are not optimal for applesauce processing in high proportions of the fruit blend.

USDA consistency results differed from those reported by Usiak and others (1995) for Idared and Rome applesauce obtained by hot-break procedure. In a previous study by our group with apple harvest season of 2009 (Chapter 2), a hot-break method was applied to obtain sauce and similar results to the literature were achieved, which did not resemble sauce obtained by cold-break..

Multiple correlation analyses (Table 3.1) indicate that applesauce consistency relates to a combination of physical and chemical factors of sauces rather than individual ones.

Yield Stress and Consistency Index

Applesauce is a dispersion of solids in liquid having yield stress and viscosity as important physical properties, which denote the applied stress required to initiate shear flow (Campanella and Pelleg, 1987) and the fluid's ability to resist motion when a shearing stress is applied (Barbosa-Canovas and others, 1996). Due to non-newtonian behavior, applesauce viscosity is described as a function of shear stress, shear rate and temperature (Sahin and Sumnu, 2008). The power law model ($\tau = K \dot{\gamma}^n$) has been commonly employed to describe the viscosity (τ) of applesauce (Rao, 2005; Ortega-Rivas, 2012) where n is the flow behavior index, K is the consistency index and $\dot{\gamma}$ is the shear rate being applied.

Table 3.1. Physical and chemical parameters affecting rheological properties of applesauce.

Parameter	Rheological Properties of Applesauce			
	USDA Consistency flow (cm)		Consistency Index (Pa.s)	Yield Stress (Pa)
	Sauce	Free-liquid		
MPS (μm)	0.6881	0.2527***	-0.7653***	-0.7579***
PSDS	-0.6748***	-0.307***	0.7685	0.7484
Soluble Solids ($^{\circ}\text{Brix}$)	-0.0409	0.2127***	-0.2144***	-0.1773
Titrateable Acidity (%)	0.045	-0.3933	0.135***	0.0211
pH	-0.1158	0.2248	0.0303	0.1118
Moisture (%)	0.1715***	-0.0067	-0.124***	-0.1172*
Calcium (ppm)	0.0744	0.1023	-0.098	-0.0134
Starch (%)	0.4643***	-0.2467***	-0.3118*	-0.4235**
AIR (%)	0.0672	-0.5239***	0.1706	0.0292
TSP (%)	-0.221	-0.4397	0.4875	0.4553
PDM (%)	0.221***	0.2362*	-0.2020	-0.1728

Correlation coefficients (r)[§] of rheological properties of applesauce (USDA consistency - sauce and free-liquid flow; consistency index and yield stress) and physical and chemical parameters assessed: mean particle size (MPS) and particle size distribution span (PSDS); soluble solids; titrateable acidity; pH; moisture; calcium; starch, alcohol insoluble residue (AIR), total soluble pectin (TSP) and pectin degree of methoxylation (PDM). Sauce was made monthly from 5 apple varieties (Crispin, Idared, Jonagold, Rhode Island Greening and Rome Beauty) stored immediately after harvest at 1 °C and 95% relative humidity over 5 months of storage and 2 harvest years ($n = 240$).

[§]Significance of r : * $p \leq 0.05$; ** $p \leq 0.01$; and *** $p \leq 0.001$.

All samples were shear-thinning ($n < 1$), and the power law model described applesauce rheology with confidence of fit $\geq 90\%$ for all samples. Applesauce yield stress and consistency index had high positive linear correlation ($R^2 = 0.94$), thus only consistency index results are presented in Figure 3.3.

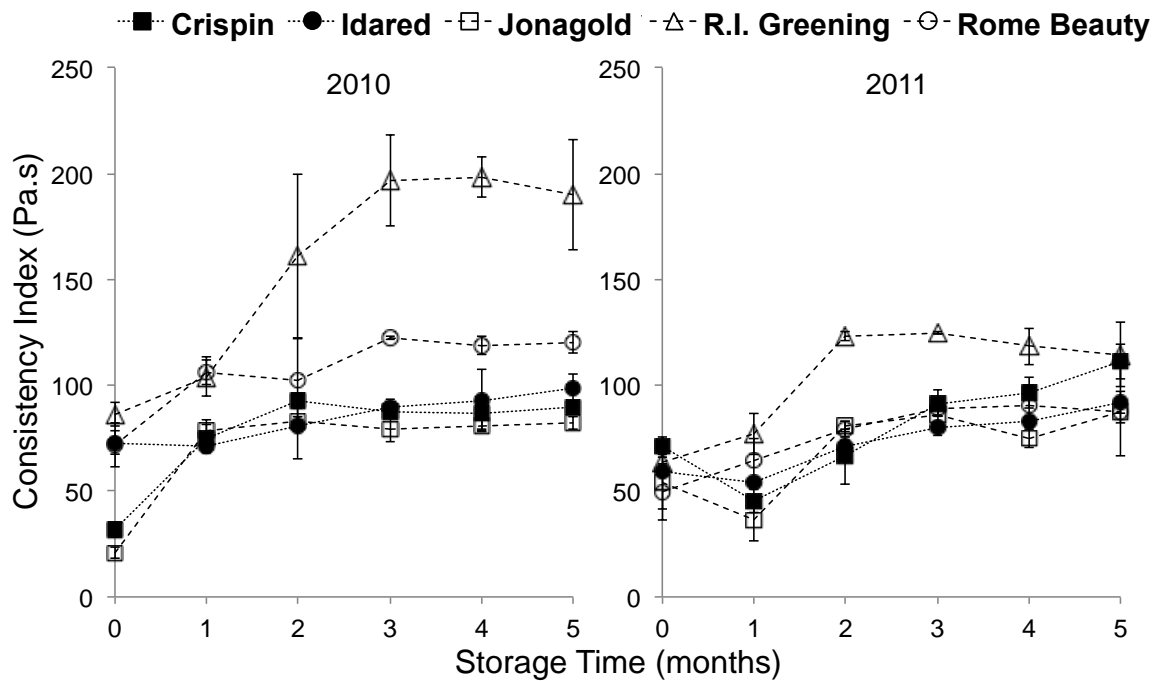


Figure 3.3 – Consistency index of applesauce made monthly from apples harvested in 2010 and 2011 stored at 1 °C and 95% relative humidity (CS) over 5 months of post-harvest storage, over 2 harvest years.

Range of results for yield stress and consistency index (19 – 211 Pa and 18 – 217 Pa.s, respectively) was comparable to those previously reported in the literature (31 – 87 Pa and 7 – 50 Pa.s, respectively) (Barbosa-Canovas and Peleg, 1983; Rao and others, 1986; Qiu and Rao, 1988; Shijvens and others, 1998), with exception of R.I. Greening, the thickest sauce studied by our group. Other minor differences in range of values observed can be attributed to applesauce processing procedures, used such as adjustment of soluble solids to up to 20 °Brix, to the different rheological assessment methods

applied and variations inherent to horticultural products due to varietal and seasonal effects.

Yield stress and consistency index were affected by harvest year and fruit variety, significantly improving with 2-3 months of storage time ($p \leq 0.05$) as a general trend. Negative linear correlation was found for sauce flow with yield stress and consistency index ($R^2 = 0.63$ and 0.62 respectively) but not for free-liquid flow indicating that sauce rheology describes only partially the differences observed in consistency, especially those related to the flow of the body of sauce. Main factors affecting applesauce yield stress and consistency index were found to be related to those affecting applesauce sauce flow (Table 3.1), notably mean particle size (MPS) and moisture. The effect of mean particle size and pulp content on hot-break applesauce has been previously reported by Qiu and Rao (1988) and seems to hold for sauce obtained by the cold-break method.

Physical and Chemical Parameters Affecting Rheological Properties of Applesauce

Mean Particle Size (MPS) and Particle Size Distribution Span (PSDS)

Mohr (1973 and 1989) reported that apple cultivar is the main factor for particle size distribution in applesauce, which is also influenced by fruit maturity at harvest, ripening, storage conditions and processing procedures. The author additionally suggested that, although single values might be used to represent the distribution of particles such average diameter or mean particle size (MPS), sometimes the full distribution is more informative. We additionally assessed the effect of particle size distribution span (PSDS), a measure of the width of the distribution.

Harvest year, apple variety and fruit ripening significantly affected particle size distribution, MPS and PSDS ($p \leq 0.05$). Both parameters were significant factors for applesauce rheological properties ($p \leq 0.001$). Overall, MPS decreased over storage at different rates based on variety significantly affecting applesauce consistency index, yield stress and free-liquid flow as PSDS increased proportionally significantly affecting sauce and free-liquid flow (Tables 3.1 and 3.2). Applesauce having smaller MPS and higher PSDS had better consistency (lower USDA consistency sauce and free-liquid flow), higher yield stress and consistency index, indicating a stronger structure, more resistant to flow. The observed effect of PSDS on applesauce consistency could be due to increased particle-to-particle interaction with increased distribution of sizes, allowing particles to pack together; as well as to larger surface area of particles to interact with the liquid phase of the dispersion, preventing it from running out of the body of sauce.

Table 3.2. Physical and chemical parameters affecting applesauce rheological properties: mean particle size (MPS); particle size distribution span (PSDS); alcohol insoluble residue (AIR); total soluble pectin (TSP) and pectin degree of methoxylation (PDM) of sauce made from apples harvested in 2010 and 2011 stored at 1 °C and 95% relative humidity over 5 months.

Parameter	Apple Variety	Harvest Year					
		2010			2011		
		Apple Storage Time (months)			Apple Storage Time (months)		
		0	3	5	0	3	5
MPS (µm)	Crispin	875 ± 7 ^a	848 ± 4 ^b	838 ± 6 ^b	985 ± 17 ^a	820 ± 10 ^c	893 ± 45 ^b
	Idared	885 ± 5 ^a	863 ± 13 ^b	797 ± 6 ^c	1019 ± 11 ^a	842 ± 15 ^b	817 ± 5 ^c
	Jonagold	888 ± 12 ^a	781 ± 14 ^b	768 ± 14 ^b	918 ± 7 ^a	843 ± 13 ^c	868 ± 14 ^b
	R.I. Greening	910 ± 7 ^a	617 ± 29 ^b	564 ± 30 ^b	909 ± 19 ^a	747 ± 11 ^b	771 ± 19 ^b
	Rome Beauty	894 ± 10 ^a	663 ± 5 ^b	647 ± 10 ^b	1051 ± 14 ^a	723 ± 12 ^b	746 ± 12 ^b
PSDS	Crispin	1.42 ± 0.07 ^a	1.44 ± 0.01 ^a	1.46 ± 0.02 ^a	1.33 ± 0.10 ^b	1.51 ± 0.02 ^a	1.39 ± 0.06 ^{ab}
	Idared	1.34 ± 0.03 ^c	1.41 ± 0.03 ^b	1.54 ± 0.01 ^a	1.18 ± 0.05 ^c	1.43 ± 0.02 ^b	1.54 ± 0.02 ^a
	Jonagold	1.40 ± 0.02 ^b	1.58 ± 0.02 ^a	1.61 ± 0.02 ^a	1.28 ± 0.01 ^c	1.44 ± 0.01 ^a	1.40 ± 0.01 ^b
	R.I. Greening	1.31 ± 0.01 ^c	1.95 ± 0.03 ^b	2.15 ± 0.10 ^a	1.50 ± 0.05 ^b	1.69 ± 0.02 ^a	1.64 ± 0.04 ^a
	Rome Beauty	1.33 ± 0.02 ^c	1.79 ± 0.04 ^b	1.86 ± 0.04 ^a	1.04 ± 0.03 ^b	1.70 ± 0.02 ^a	1.70 ± 0.03 ^a
AIR (%)	Crispin	1.84 ± 0.15 ^a	2.03 ± 0.04 ^a	1.98 ± 0.14 ^a	-	2.48 ± 0.19 ^a	2.26 ± 0.19 ^a
	Idared	2.86 ± 0.44 ^a	1.80 ± 0.01 ^b	1.82 ± 0.03 ^b	3.09 ± 0.02 ^a	2.13 ± 0.10 ^c	2.34 ± 0.02 ^b
	Jonagold	1.74 ± 0.17 ^c	3.58 ± 0.17 ^a	2.98 ± 0.44 ^b	2.17 ± 0.04 ^{ab}	2.71 ± 0.53 ^a	2.02 ± 0.12 ^b
	R.I. Greening	3.70 ± 0.10 ^a	2.95 ± 0.45 ^b	3.63 ± 0.46 ^{ab}	5.46 ± 0.26 ^a	2.68 ± 0.04 ^b	2.76 ± 0.05 ^b
	Rome Beauty	2.55 ± 0.27 ^a	2.59 ± 0.13 ^a	2.38 ± 0.26 ^a	2.59 ± 0.20 ^a	2.02 ± 0.25 ^b	2.14 ± 0.06 ^b
TSP (%)	Crispin	0.16 ± 0.01 ^b	0.28 ± 0.01 ^a	0.28 ± 0.04 ^a	-	0.24 ± 0.01 ^a	0.20 ± 0.02 ^a
	Idared	0.23 ± 0.04 ^a	0.26 ± 0.01 ^a	0.26 ± 0.01 ^a	0.28 ± 0.04 ^{ab}	0.32 ± 0.05 ^a	0.23 ± 0.04 ^b
	Jonagold	0.21 ± 0.01 ^c	0.52 ± 0.01 ^a	0.31 ± 0.04 ^b	0.29 ± 0.01 ^b	0.45 ± 0.08 ^a	0.15 ± 0.02 ^c
	R.I. Greening	0.27 ± 0.01 ^b	0.42 ± 0.05 ^a	0.47 ± 0.05 ^a	0.30 ± 0.02 ^{ab}	0.32 ± 0.04 ^a	0.27 ± 0.02 ^b
	Rome Beauty	0.25 ± 0.01 ^b	0.40 ± 0.03 ^a	0.35 ± 0.04 ^b	0.29 ± 0.04 ^a	0.26 ± 0.01 ^a	0.24 ± 0.07 ^a
PDM (%)	Crispin	63.4 ± 9.5 ^a	74.5 ± 5.4 ^a	72.7 ± 3.4 ^a	-	55.4 ± 1.0 ^a	55.8 ± 3.2 ^a
	Idared	63.8 ± 1.0 ^b	74.8 ± 2.6 ^a	72.5 ± 0.7 ^a	41.4 ± 0.9 ^b	48.0 ± 0.2 ^b	65.6 ± 9.1 ^a
	Jonagold	73.8 ± 5.1 ^a	76.4 ± 0.4 ^a	74.8 ± 0.3 ^a	42.4 ± 3.6 ^b	64.8 ± 2.1 ^a	73.1 ± 6.2 ^a
	R.I. Greening	56.6 ± 3.0 ^a	47.2 ± 0.9 ^c	51.1 ± 1.2 ^b	67.4 ± 10.0 ^a	63.2 ± 7.0 ^{ab}	51.7 ± 0.4 ^b
	Rome Beauty	41.7 ± 6.2 ^a	44.1 ± 8.2 ^a	36.7 ± 2.7 ^a	45.4 ± 0.6 ^a	42.3 ± 5.4 ^a	49.8 ± 10.3 ^a

Figure 3.4 (a, b, c and d) illustrates overall particle size distribution changes in applesauce with apple storage time (ST) – ST = 0 and 5 months for 2010 and 2011 harvests – for 3 varieties representative of the most important trends observed for applesauce consistency. Best consistency applesauce with progress of fruit storage (lower sauce and free-liquid flow) was achieved mostly by R.I. Greening and Rome, varieties showing greater MPS reduction and increased PSDS over storage.

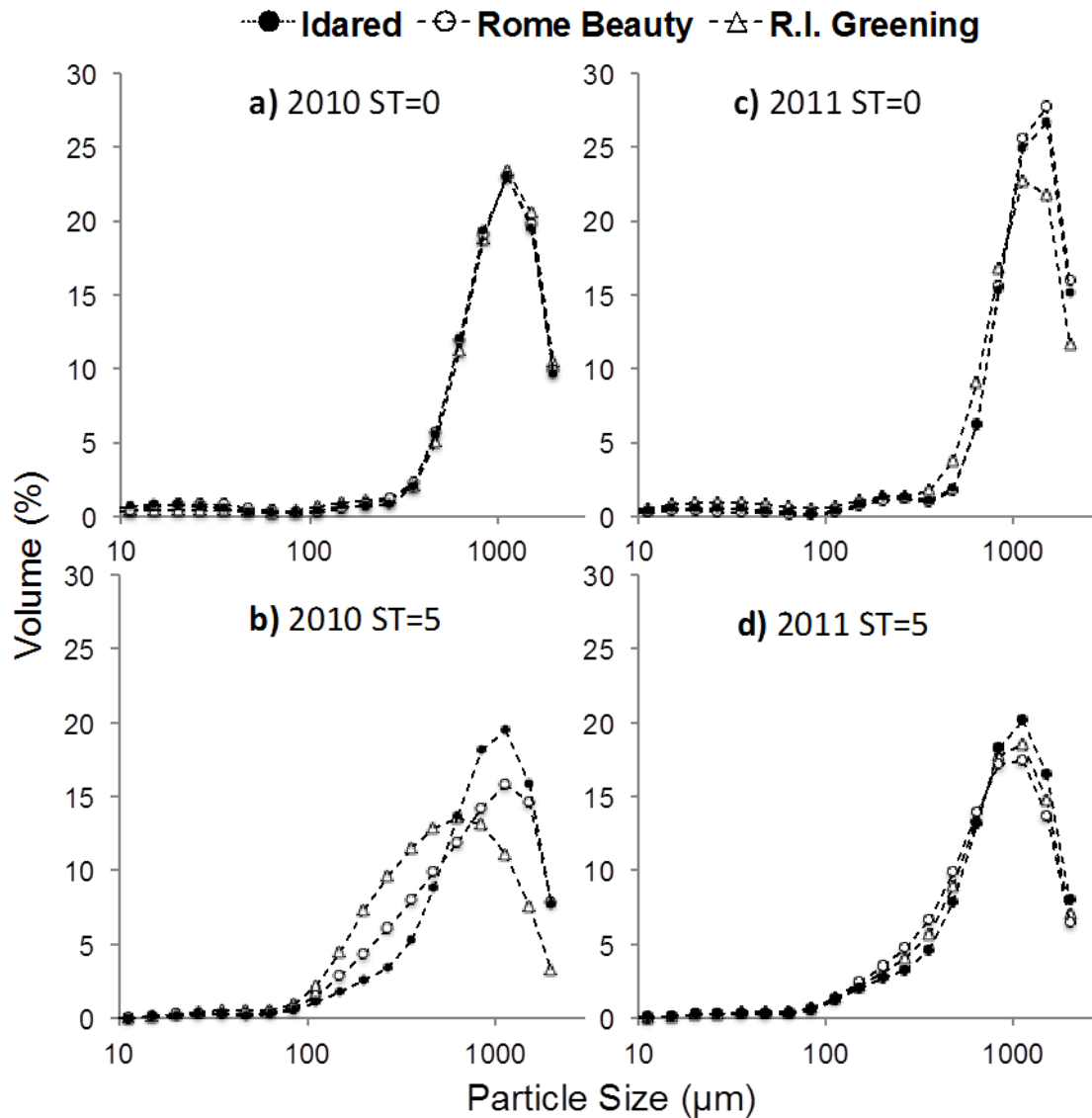


Figure 3.4 – Changes in particle size distribution of applesauce made from apples stored at 1 °C and 95% relative humidity over 5 months of apple storage time (ST), over 2 harvest years.

Moisture

Applesauce moisture was variety dependent ranging 86.1-90.1 and 83.5-91.1% in 2010 and 2011 respectively, and significantly affected sauce flow, yield stress and consistency index (Table 3.1) in agreement with previous reports on the effect of pulp content on rheological properties of sauce (Beresovsky and others, 1995; Metzner, 1985; Tanglertpaibul and Rao, 1987; Qiu and Rao, 1988).

Starch

Starch is present as energy storage in most fruit tissues. As the fruit matures, starch begins to hydrolyze into sugars (Brookfield and others, 1997). Traceable amounts of starch can be left in fruits upon harvesting (Belitz, 2009).

Starch content was variety dependent and significantly decreased over the first 3 months of fruit CS storage becoming negligible ($\leq 0.01\%$) thereafter (Figure 3.5). Similar results have been previously reported in the literature for apples (Smock and Neubert, 1950; Fischer and Amado, 1994). Starch content was a significant factor for all rheological properties assessed, notably for USDA free-liquid flow, as it seems to explain significant differences observed in the liquid-separation of sauces at the beginning of both harvest years ($ST = 0$) for all varieties; while for other rheological properties it might have stood out as a significant factor due to starch degradation occurring when other post-harvest fruit ripening-related changes were taking place affecting sauce composition (2-3 months of fruit CS storage), leading to improved yield stress, consistency and sauce flow with progress of fruit storage.

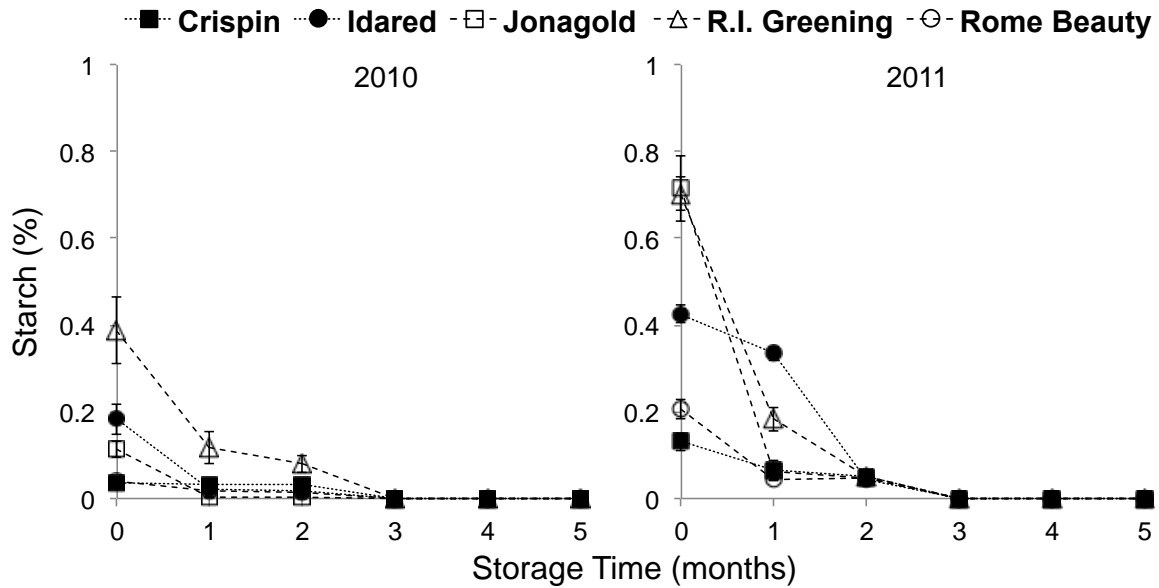


Figure 3.5 – Changes in starch content of applesauce made monthly from apples stored at 1 °C and 95% relative humidity over 5 months of storage time, over 2 harvest years.

Calcium

Calcium has long been thought to be important as a crosslinking agent for polygalacturonide chains in plant cell walls (Knee, 1973). The investigation of calcium content in varietal applesauce is important due to its ability to form gels with pectins as the degree of esterification of pectin decreases (Van Buren, 1991). Calcium was not significantly different across harvest years, apple variety or storage time ranging 18-37 ppm and was not a significant factor for applesauce consistency. Our results are in agreement with studies from Sams and Conway (1993) who reported that free liquid in applesauce made from calcium treated apples was not affected as calcium concentration increased. Apples, apple juice and pulp are reported to have between 20-130 ppm on a fresh weight basis (Perring, 1974; Nour and others, 2010). The low range of results observed in our study may be due to the fact that calcium was measured in applesauce serum.

Alcohol Insoluble Residue (AIR)

The residue after alcohol wash – alcohol insoluble residue – (AIR) consists of polysaccharides such as pectic substances together with a small amount with proteins Ladaniya (2008). According to Stephen and Williams (2006), they can prevent sedimentation and liquid separation in food systems due to their thickening properties.

AIR was a significant factor for applesauce free-liquid flow, being strongly dependent on harvest year, apple variety and CS storage time ($p \leq 0.0001$) ranging from 1.59-5.68% (Table 3.2). It was overall higher for Greening applesauce (averaging 3.43 and 3.64% in 2010 and 2011 respectively) and showed less marked differences for other varieties (averaging 1.95-2.79 and 2.25-2.52% in 2010 and 2011 respectively). The range of results was comparable to those found in the literature for apples and applesauce (1.76-5.48%) (De Vries, 1981; Fischer and others, 1994; Colin-Henrion, 2009).

AIR was a significant factor for sauce free-liquid flow (Table 3.1) and changed according to differences in starch and TSP of sauce and their interaction ($p \leq 0.05$), indicating that AIR quantification could potentially serve as a quick estimator of those compounds for applesauce manufacturers for product quality control due to the methodology's simplicity and few equipment requirements.

Total Soluble Pectin (TSP) and Pectin Degree of Methoxylation (PDM)

Total soluble pectin (TSP) content in applesauce (Table 3.1) was also dependent on apple variety showing harvest year effects and overall increasing slightly with progress of apple storage and remaining stable or decreasing slightly towards the end of

storage (Table 3.2). This behavior has long been reported as a natural process of apple ripening related to the depolymerization of pectin fractions from insoluble to soluble and eventual degradation into non pectic materials (Smock and Neubert, 1950; Massey and others, 1964; Knee and Bartley, 1981; Fischer, 1991). Range of results (0.13 – 0.54%) and trends observed were similar to those reported in the literature (0.17 – 0.55%) for apples, with apple storage time, and for varietal applesauce (McClendon and others, 1959; De Vries, 1981; Lo Scalzo and others, 2005; Vanoli and others, 2009; Le Bourvellec and others, 2011). In our statistical model, TSP was not a significant factor for sauce free-liquid flow as in previous reports by Toldby and Wiley (1962) probably due to stronger effect of AIR, which comprises both soluble and insoluble pectin fractions other than starch and a small amount of proteins, all of which can prevent liquid-separation of the structure. TSP was nonetheless overall higher in thicker sauce, which had higher yield stress and consistency index readings, and lower sauce flow and liquid-separation (Table 3.1). This is consistent with general knowledge about functional properties of pectins such as their ability to yield gels in the presence of calcium and to increase the viscosity of systems (Phatak and others, 1988; Michel and others, 1985; Arslan and Togrul, 1996).

Pectin degree of methoxylation (PDM) was further assessed due to potential effects of this parameter to product consistency, since it signals the ability of pectin to yield gels in the presence of calcium. As the degree of methoxylation of pectin decreases, carboxylic acid groups along the polygalacturonic acid backbone of pectin become available to bind with calcium ions forming a cross-link structure described by Kertz (1952) as the egg-box model.

PDM in applesauce was dependent on apple variety showing harvest year effects and being, in general, stable with progress of apple storage or decreasing towards the end of storage (Table 3.2). It was a significant factor for sauce and free-liquid flow being overall lower in thicker sauce showing lower sauce flow and less liquid separation (Table 3.1). Our results (42-76%) were comparable to those reported in the literature (47-88%) for apple varieties, with progress of storage time and varietal applesauce (De Vries and others, 1984; Klein and others, 1995; Johnston and others, 2002; Rascón-Chu and others, 2009; Le Bourvellec and others, 2011).

The effect of PDM to rheological properties of applesauce observed in our study is in agreement with previous findings by Usiak and others (1995) who reported improvements of applesauce consistency by processing product through a low-temperature blanch in the optimal temperature range for the activity of the enzyme pectin methyl-esterase (PME), thus a decrease PDM was expected although not measured.

Conclusions

Rheological properties of cold-break applesauce were dependent on apple variety, storage time and harvest year. The main effects can be explained by differences in physical and chemical parameters related to varietal composition of sauce and post-harvest fruit ripening over storage. Sauce of improved rheological properties for all varieties was achieved after fruit had been stored for 2-3 months in cold storage due to ripening changes leading to sauce of lower MPS and increased PSDS, parameters that could potentially be tracked by applesauce manufacturers for quality control of product consistency. Free-liquid flow of sauces was lower in sauce having high levels of starch in

the beginning of the harvest season and higher AIR and lower PDM throughout fruit storage. AIR changed according to changes in starch and TSP of sauces and could be additionally tracked for quality control purposes of product consistency by applesauce manufacturers due to the methodology simplicity and few equipment requirements.

References

- Ahmed A, Labavitch, J. 1977. A simplified method for accurate determination of cell wall uronide content. *J Food Biochem* 1:361–365.
- Anthon, G. E., & Barrett, D. M. 2004. Comparison of three colorimetric reagents for the determination of methanol with alcohol oxidase. Application to the assay of pectin methylesterase. *J Agric Food Chem* 52: 3749–3753.
- Anthon GE, Barrett DM. 2008. Combined enzymatic and colorimetric method for determining the uronic acid and methylester content of pectin: Application to tomato products. *Food Chem* 110(1):239-47.
- AOAC International. 2000. *Official Methods of Analysis of AOAC International*. 17th ed. Arlington: AOAC International. 2200 p.
- Arslan N, Toğrul H. 1996. Filtration of pectin extract from grapefruit peel and viscosity of pectin solutions. *J Food Eng* 27(2):191-201.
- Barbosa-Cánovas GV, Peleg M. 1983. Flow parameters of selected commercial semi-liquid food products. *J Texture Stud* 14(3):213-34.
- Belitz HD, Grosch W, Schieberle P. 2009. *Food Chemistry*. 4th ed. New York: Springer. 1070 p.
- Beresovsky N, Kopelman IJ, Mizrahi S. 1995. The role of pulp interparticle interaction in determining tomato juice viscosity. *J Food Process Preserv* 19(2):133-46.
- Blumenkrantz, N., & Asboe-Hansen, G. 1973. New method for quantitative determination of uronic acids. *Anal Biochem* 54: 484–489.
- Brookfield P, Murphy P, Harker R, MacRae E. 1997. Starch degradation and starch pattern indices; interpretation and relationship to maturity. *Postharvest Biol. Technol* 11(1):23-30.

- Brownleader M, Jackson P, Mobasheri A, Pantelides A, Sumar S, Trevan M, Dey P. 1999. Molecular aspects of cell wall modifications during fruit ripening. *Crit Rev Food Sci Nutr* 39(2):149-64.
- Burg SP. 2004. *Postharvest Physiology and Hypobaric Storage of Fresh Produce*. Cambridge: CABI Publishing. 654 p.
- Calvin L, Martin P. 2011. *The U. S. Produce Industry and Labor: Facing the Future in a Global Economy*, ERR-106, U.S. Department of Agriculture, Economic Research Service, November 2010. DIANE Publishing. 57p.
- Campanella OH, Peleg M. 1987. Determination of the yield stress of semi-liquid foods from squeezing flow data. *J Food Sci* 52(1):214-5.
- Chin, L. H., Ali, Z. M., & Lazan, H. 1999. Cell wall modifications, degrading enzymes and softening of carambola fruit during ripening. *J Exp Bot* 50(335): 767–775.
- Colin-Henrion M, Mehinagic E, Renard CMGC, Richomme P, Jourjon F. 2009. From apple to applesauce: Processing effects on dietary fibres and cell wall polysaccharides. *Food Chem* 117(2):254-60.
- De Vries JA, Voragen AGJ, Rombouts FM, Pilnik W. 1981. Extraction and purification of pectins from Alcohol Insoluble Solids from ripe and unripe apples. *Carbohydr.Polym* 1(2):117-27.
- De Vries JA, Rombouts FM, Voragen AGJ, Pilnik W. 1984. Comparison of the structural features of apple and citrus pectic substances. *Carbohydr.Polym* 4(2):89-101.
- FDA: 21CFR145.110 – Canned Applesauce [Internet]. Silver Spring, MD: U.S. Food and Drug Administration [Accessed 2012 Sep 17]. Available from: <http://www.accessdata.fda.gov/scripts/cdrh/cfdocs/cfcfr/CFRSearch.cfm?fr=145.110>.
- Fischer RL, Bennett A. 1991. Role of cell wall hydrolases in fruit ripening. *Annual review of plant biology* 42(1):675-703.
- Fischer M, Arrigoni E, Amadò R. 1994. Changes in the pectic substances of apples during development and postharvest ripening. Part 2: Analysis of the pectic fractions. *Carbohydr.Polym* 25(3):167-75.
- Goulao LF, Oliveira CM. 2008. Cell wall modifications during fruit ripening: when a fruit is not the fruit. *Trends Food Sci.Technol* 19(1):4-25.
- Johnston J, Hewett E, Hertog M. 2002. Postharvest softening of apple (*Malus domestica*) fruit: A review. *N Z J Crop Hortic Sci* 30(3):145-60.

- Kertesz ZI. 1952. The pectic substances. New York: Interscience Publishers, Inc. 628 p.
- Knee M. 1973. Polysaccharide changes in cell walls of ripening apples. *Phytochemistry* 12(7):1543-9.
- Knee M, Bartley IM. 1981. Composition and metabolism of cell-wall polysaccharides in ripening fruit. In: *Recent Advances in Biochemistry of Fruits and Vegetables*. New York: Academic Press. P 133-148.
- Klein JD, Hanzon J, Irwin PL, Shalom NB, Luria S. 1995. Pectin esterase activity and pectin methyl esterification in heated golden delicious apples. *Phytochemistry* 39(3):491-494.
- Konopacka D, Plochanski WJ. 2004. Effect of storage conditions on the relationship between apple firmness and texture acceptability. *Postharvest Biol Technol* 32(2):205-11.
- La Belle RL. 1981. Apple quality characteristics as related to various processed products. In: R. Teranishi and H. Barrera-Benitez. *Quality of selected fruits and vegetables of North America*. ACS Symposium Series. Washington: American Chemical Society. p. 61-76.
- Ladaniya MS. 2008. *Citrus fruit: biology, technology and evaluation*. San Diego: Academic Press. 558p.
- Lanza JJR, Kramer A. 1967. Objective measurement of graininess in apple sauce. *P Am Soc Hort Sci* 90:491-497.
- Le Bourvellec C, Bouzerzour K, Ginies C, Regis S, Plé Y, Renard CMGC. 2011. Phenolic and polysaccharidic composition of applesauce is close to that of apple flesh. *Journal of Food Composition and Analysis* 24(4-5):537-47.
- Lo Scalzo R, Forni E, Lupi D, Giudetti G, Testoni A. 2005. Changes of pectic composition of 'annurca' apple fruit after storage. *Food Chem* 93(3):521-530.
- Louis M, Massey JR. 1989. Harvesting, Storing and Handling Processing Apples. In: Downing DL. *Processed apple products*. New York: Van Nostrand Reinhold. P 215-238.
- Massey Jr. LM. 1989. Harvesting, storing and handling processing apples. In: Downing DL. *Processed apple products*. New York: Van Nostrand Reinhold. P 31-51.
- McClendon JH, Woodmansee CW, Somers GF. 1959. On the Occurrence of Free-Galacturonic Acid in Apples and Tomatoes. *Plant Physiol* 34(4):389-91.

- McLellan MR, Massey LM. 1984. Effect of Postharvest Storage and Ripening of Apples on the Sensory Quality of Processed Applesauce. *J. Food Sci* 49(5):1323-6.
- Metzner AB. 1985. Rheology of suspensions in polymeric liquids. *J Rheol* 29(6): 739-775.
- Mohr WP. 1973. Applesauce grain. *J Texture Stud* 4(2):263-8.
- Mohr WP. 1989. Influence of cultivar, fruit maturity and fruit anatomy on apple sauce particle size and texture. *Int J Food Sci Tech* 24(4):403-13.
- New York Apple Association: New York apples fast facts [Internet]. Victor, NY: New York Apple Association [Accessed 2011 Jun 13]. Available from: <http://www.nyapplecountry.com/fastfacts.htm>.
- Nour V, Trandafir I, Ionica ME. 2010. Compositional characteristics of fruits of several apple (*Malus domestica* Borkh.) cultivars. *Not Bot Hort Agrobot Cluj* 38 (3): 228-233.
- Ortega-Rivas E. 2012. Non-thermal food engineering operations. New York: Springer. 375 p.
- Phatak L, Chang CK, Brown G. 1988. Isolation and characterization of pectin in sugar-beet pulp. *J Food Sci* 53(3):830-3.
- Perring MA. 1974. The chemical composition of apples. XI. An extraction technique suitable for the rapid determination of calcium, but not potassium and magnesium, in the fruit. *J Sci Food Agric* 25(3): 237–245.
- Perring MA. 1989. Apple fruit quality in relation to fruit chemical composition. *Acta Hort* 258:365-372.
- Qiu C, Rao MA. 1988. Role of pulp content and particle size in yield stress of apple sauce. *J Food Sci* 53(4):1165-1170.
- Rao MA, Cooley HJ, Nogueira JN, McLellan MR. 1986. Rheology of apple sauce: effect of apple cultivar, firmness, and processing parameters. *J Food Sci* 51(1):176-179.
- Rao MA. 2005. Rheological properties of fluid foods. In: Rao MA, Rizvi SSH, Datta AK. *Engineering properties of foods*. New York: CRC Press. p 41-98.
- Rascón-Chu A, Martínez-López AL, Carvajal-Millán E, Ponce de León-Renova NE, Márquez-Escalante JA, Romo-Chacón A. 2009. Pectin from low quality 'golden delicious' apples: composition and gelling capability. *Food Chem* 116(1):101-103.

- Sams CE, Conway WS. 1993. Postharvest calcium infiltration improves fresh and processing quality of apples. *Acta Hort* 326:123-130.
- Schijvens EPHM, Van Vliet T, Van Dijk C. 1998. Effect of Processing Conditions on the composition and rheological properties of applesauce. *J Texture Stud* 29(2):123-143.
- Stephen AM. 1995. Food polysaccharides and their applications. CRC press, New York. 752 p.
- Sila, D. N., Doungra, E., Smout, C., Van Loey, A., & Hendrickx, M. (2006). Pectin fraction interconversions: Insight into understanding texture evolution of thermally processed carrots. *Journal of Agricultural and Food Chemistry*, 54(22), 8471–8479.
- Smock RM, Neubert AM. 1950. Apples and apple products. New York: Interscience Publishers. P 486.
- Stephen AM. 1995. Food polysaccharides and their applications. CRC press, New York. 752 p.
- Tanglertpaibul T, Rao MA. 1987. Flow properties of tomato concentrates: effect of serum viscosity and pulp content. *J Food Sci* 52(2): 318–321.
- Toldby V, Willey R. 1962. Liquid-solids separation, a problem in processed applesauce. *J Am Soc Hortic Sci* 81:78-90.
- USDA: Grading Manual for Canned Applesauce [Internet]. Washington, D.C.: United States Department of Agriculture [Accessed 2009 Sep 19]. Available from: <http://www.usda.gov>.
- USDA: U.S. Apple Statistics [Internet]. National Agricultural Statistics Service, Cold Storage Annual Summary, various issues. Washington, D.C.: United States Department of Agriculture [Accessed 2012 Mar 31]. Available from: <http://usda.mannlib.cornell.edu/MannUsda/viewDocumentInfo.do?documentID=1825>.
- Usiak AMG, Bourne MC, Rao MA. 1995. Blanch temperature/time effects on rheological properties of applesauce. *J.Food Sci.* 60(6):1289-1291.
- Van Buren JP. 1991. Function of pectin in plant tissue structure and firmness. In: Walter RH. *The chemistry and technology of pectin*. San Diego: Academic Press. p 1-22.
- Wiley RC, Binkley CR. 1989. Applesauce and other canned apple products. In: Downing DL. *Processed apple products*. New York: Van Nostrand Reinhold. P 215-23.

- Vanoli M, Zerbini PE, Spinelli L, Torricelli A, Rizzolo A. 2009. Polyuronide content and correlation to optical properties measured by time-resolved reflectance spectroscopy in 'Jonagored' apples stored in normal and controlled atmosphere. *Food Chem* 115(4):1450-1457.
- Varela P, Salvador A, Fiszman S. 2007. Changes in apple tissue with storage time: Rheological, textural and microstructural analyses. *J.Food Eng* 78(2):622-9.
- Watkins, C.B. 2003. Principles and practices of postharvest handling and stress. In: *Apples: Crop Physiology, Production and Uses*. Feree, D. and I.J. Warrington (eds) Chapt. 23, CAB Pub. pp. 585-614.

CHAPTER 4:
COLD-BREAK APPLESAUCE RHEOLOGICAL PROPERTIES IMPROVE
WITH CONTROLLED POST-HARVEST FRUIT RIPENING

ABSTRACT: Cold-break applesauce manufacturers face challenges in achieving products of optimal consistency when processing newly harvested fruit. Four freshly-harvested apple varieties (Cortland, Empire, Golden Delicious and McIntosh) were stored at 2 different conditions at 95% relative humidity (RH) – 1 °C for up to 5 months (cold storage – CS, control group) and at 10 °C for up to 4 weeks for accelerated post-harvest ripening – for 2 harvest years to assess the effect of fruit storage on rheological properties of applesauce and potential benefit of accelerated post-harvest ripening practices. Sauce was obtained monthly and weekly, respectively, following a cold-break procedure. Apples were analyzed for ripening indicators (firmness, pH, acidity, soluble solids); and applesauce for rheological properties (USDA consistency, yield stress; consistency index); particle size distribution, mean particle size (MPS) and particle size distribution span; moisture; calcium and starch. Results were analyzed by ANOVA and significant differences among means determined by Tukey's test ($p \leq 0.05$). Harvest season, apple variety and storage time under different storage conditions were all significant factors for applesauce rheological properties ($p\text{-value} \leq 0.05$). Significant improvements in sauce rheology were found after 3 months at 1 °C and after 3 weeks at 10 °C. Fruit storage at 10 °C for 3 weeks produced sauce of MPS similar to that of fruit stored at 1 °C for 3 months, a significant factor for applesauce rheology. Further chemical analyses on applesauce made from cold stored apples – alcohol insoluble residue; total soluble pectin and pectin degree of methylation – indicate that those parameters are also important factors to explain rheological differences observed..

Key words: applesauce; consistency; rheology; storage.

Practical Application: cold-break applesauce manufacturers face challenges in achieving good consistency products early in the processing year. Accelerated post-harvest fruit ripening at uncontrolled conditions of temperature, relative humidity and time, have been empirically applied to newly harvested apples in an attempt to promote compositional changes leading to product of improved rheological properties. The efficacy of this practice is however, unconfirmed, potentially being susceptible to varietal and harvest year effects. Information about the impact of post-harvest fruit ripening and storage conditions on sauce rheological properties will provide valuable information for raw material storage management by applesauce manufacturers.

Introduction

Applesauce is an apple-based canned product made from comminuted or chopped apples defined by FDA (2012). Canned products such as applesauce were forecasted to use 12% of the total 2011 crop together with apple slices (U.S. Apple Association, 2011). Rheological properties of fluid foods such as applesauce are important for quality control and sensory evaluation (Rao, 1977). Consistency, a flow measurement related to the separation of liquid in applesauce, is an important attribute for product grading (USDA, 2009).

Most apple varieties are harvested from August to October in the U.S (Calvin and Martin, 2011). Raw material availability for processing throughout the year is achieved by the use of cold (CS) and controlled atmosphere (CA) storages – 1-4 °C at 95-98% relative humidity (RH) at normal or modified atmosphere (1-3% O₂ and 1-5% CO₂),

respectively – for up to 6 to 12 months, depending on storage conditions and varietal suitability to storage (Skog and Chu, 2003).

Additionally, apples for food manufacturing are seldom used freshly harvested. Instead, accelerated post-harvest fruit ripening is often applied to alter fruit composition to suit processing purposes. The practice consists of holding fruit (either directly from harvest or from storage) at higher temperatures than common storage (CS or CA) to accelerate the respiratory activity of apples and changes that take place with such aging (Louis and Massey, 1989). La Belle (1981) suggested that benefits of using fully matured and well-ripened apples for sauce processing are related to flavor development and textural changes. Moreover, according to cold-break manufacturers, accelerated post-harvest fruit ripening is applied to apples for sauce production in an effort to improve sauce rheological properties.

Improvements of applesauce rheological properties due to accelerated post-harvest fruit ripening could be related to physical and chemical changes in sauce composition such as mean particle size reduction due to fruit tissue softening, changes in polysaccharide composition affecting rheological properties and/or water holding capacity of the system (Qiu and Rao, 1988; Beresovsky and others, 1995; Usiak and others, 1995). Because parameter values and changes over storage are reported to occur differently in the sauce of different apple varieties (Toldby and Wiley, 1962; McLellan and Massey, 1984; Rao and others, 1986; Mohr, 1989; Le Bourvellec and others, 2011), the benefit of the practice should also be investigated on a varietal basis.

Manufacturing facilities are constantly processing a blend of apple varieties (Wiley and Binkley, 1989), often at different stages of fruit ripening. Accelerated post-

harvest fruit ripening is usually carried out by holding process stock for arbitrary periods of time outside the processing facility, exposed to variable conditions of temperature and RH, which can lead to variable results, rendering conclusions about the practice's efficacy limited.

Our objective was to assess the potential benefit of controlled post-harvest fruit ripening to rheological properties of applesauce by comparing sauce made from apples stored at 10 °C and 95% RH for up to 4 weeks to that of fruit stored at regular CS conditions for up to 5 months. Varietal and harvest year effects were accounted for by assessing sauce of 4 different varieties over 2 harvest years (2010 and 2011).

Materials and Methods

Apples

Apples (*Malus domestica* Borkh.) that included Cortland, Empire, Golden Delicious and McIntosh, were harvested from apple farms located in New York State between September and November of 2010 and 2011, and were immediately delivered to the processing pilot plant at Cornell University. Apples were kept at 10 °C and 95% RH (controlled post-harvest fruit ripening group) or CS conditions (1-4 °C and 95% RH – control group) until processing day carried out weekly or monthly (every 6-7 or 28-30 days), respectively.

Apple Maturity Indicators and Applesauce Processing

Prior to processing, apples were weighed and tested for firmness using a hand-held penetrometer model FT 327 (Wagner Instruments, Greenwich, CT). Sauce making

followed a cold-break procedure: apples (~ 15 kg) were fed through turbo extractor (1.6 mm screen, 8 mm gap, 1800 rpm; Bertocchi CX5, Bertocchi SLR., Parma, Italy) and 15% water (w/w) was added to the sauce (to simulate water pick-up by direct steam injection) which was then heated in steam kettle at 96-98 °C for 6 min and hot-filled into 8 oz glass jars. Jars were inverted for 3 min for cap sterilization, cooled in water bath and stored at 1 °C until analysis. A sample of comminuted apples (turbo extractor output) was collected and pressed through cheesecloth to obtain juice which was tested for pH using a bench-top Thermo Scientific pHmeter model Orion 3-Star (Cellomics, Pittsburgh, PA); titratable acidity (TA) – through titration with NaOH 0.1 N and recorded as % malic acid; and soluble solids – according to AOAC (2000) utilizing a bench-top refractometer model Leica Auto Abbe (Leica Inc., Buffalo, NY).

Applesauce

Applesauce yield stress and consistency index of samples were determined using a vane spindle model V-73 in a Brookfield DV-III Ultra programmable rheometer at constant temperature (25 °C) with software package RheoCalc (equipment and program from Brookfield Engineering Laboratories, INC. Middleboro, MA). Yield Stress and Consistency Index were calculated using the Casson and power law models, respectively, from shear-stress data obtained by subjecting samples to 0.5 s⁻¹ increments of shear-rate from 0.5 to 3.0 s⁻¹ upward and backward with 1 min hold at each shear-rate prior to data collection every 1-min, during a total time of 11 min. USDA consistency was measured according to the Grading Manual for Canned Applesauce (USDA, 2009) and qualitative consistency grading was assigned. The volume-based particle size distribution (PSD)

Mean particle size (MPS) and particle size distribution span (PSDS) were assessed using a Malvern laser diffraction unit model Mastersizer 2000 (Malvern Instruments Inc., Westborough, MA). MPS was calculated as the volume-based mean particle diameter ($\sum_i n_i d_i^4 / \sum_i n_i d_i^3$ where n_i is the number of particles of diameter d_i) and PSDS was calculated as width of the volume-based particle size distribution ($(d_{90th\ percentile} - d_{10th\ percentile}) / d_{50th\ percentile}$). Applesauce moisture was measured according to AOAC (2000). Applesauce pH was obtained as previously described for apple slices. Applesauce samples were centrifuged at 17000 rpm for 30 min and the supernatant (applesauce serum) was collected and stored at -10 °C until further analysis. Alcohol insoluble residue (AIR), total soluble pectin (TSP) and pectin degree of methoxylation (PDM) analysis were carried out as previously described in detail in Chapter 2. Isolation of alcohol insoluble residue (AIR) was carried out according to Mcfeeters & Armstrong (1984) and reported as % AIR in applesauce. AIR was ground and pulverized for extraction of water- and chelator-soluble pectin fractions (WSP and CSP, respectively). The WSP and CSP fractions were obtained following the procedures by Sila and others (2006) and Chin and others (1999), respectively. Each fraction was analyzed for Galacturonic acid (GalA) and methanol for determining pectin content (as GalA equivalent) and pectin degree of methoxylation (as the ratio of the molar amount of methanol esters to the molar amount of galacturonic acid residues). The GalA content in pectin fractions was determined by hydrolysis in H₂SO₄/ tetraborate solution (0.0125 M solution of sodium tetraborate in concentrated sulfuric acid) as described by Ahmed and Labavitch (1977) with subsequent colorimetric determination according to Blumenkrantz and Asboe-Hansen (1973) by using a Barnstead Turner SP830 Spectrophotometer

(Barnstead International, Dubuque, IA). The methanol concentration was determined by alkaline hydrolysis of 1 volume of sample in 2 volumes of 0.5 M NaOH and subsequent incubation at room temperature for 1 hour followed by neutralization with 1 volume of 1 M HCl according to Anthon and Barrett (2008). The amount of methanol was determined using alcohol oxidase and Purpald as described by Anthon and Barrett (2004). WSP and CSP were proportionally combined as fractions of applesauce AIR in order to obtain values for TSP and PDM of sauces. Additionally, applesauce samples were centrifuged at 17000 rpm for 30 min and the supernatant (applesauce serum) was collected through filtration also using Whatman filter paper 55 mm. The serum was stored at -10 °C until further analysis. Applesauce serum titratable acidity was obtained as previously described for apples. Calcium concentration in applesauce serum was determined using Calcium-Arsenazo quantification kit (BEN Biochemical Enterprise, Milano, Italy). Starch analysis was performed through iodine-iodide 0.01 N reaction measuring absorbance at 570 nm referring to a standard curve of known starch concentrations in a Turner spectrophotometer model Barnstead SP-830 (Turner Biosystems, Dubuque, IA) reported as % starch (g/100 ml).

Statistical Analysis

Two batches of apples were processed into sauce generating two replicates, resulting in a total of 4 samples for each experimental point. Measurement for all experimental units was conducted in duplicate or triplicate and results were expressed as means and standard deviations. Data was analyzed by ANOVA and significant differences among means adopting a 95 % confidence interval ($p \leq 0.05$) were

determined by Tukey's test using JMP® 9.0 statistical software (SAS institute Inc., Cary, NC).

Results and Discussion

Apple Ripening Indicators

Apple ripening indicators (firmness, pH, titratable acidity and soluble solids) were significantly affected by harvest year, apple variety, and storage time as extensively reported in the literature (Smock and Neubert, 1950; Massey Jr., 1989; La Belle, 1981). Fruit storage at 10 °C and 95% RH for 3 weeks was able to reproduced changes in apple firmness occurring to fruit stored at 1 °C and 95% RH for 3 months for most varieties studied in both harvest years (Figure 4.1). Similar reproducibility was also observed for other ripening indicators – apple pH, titratable acidity and soluble solids ranging 3.11 – 3.77 and 3.14 – 3.67; 0.730 – 0.414 and 0.729 – 0.227; and 10.01 – 15.15 and 10.70 – 15.16 in 2010 and 2011 respectively –, suggesting that accelerated post-harvest fruit ripening at these conditions is an efficient way to promote changes occurring to apples, and that tracking of ripening indicators might help to determine best time for processing.

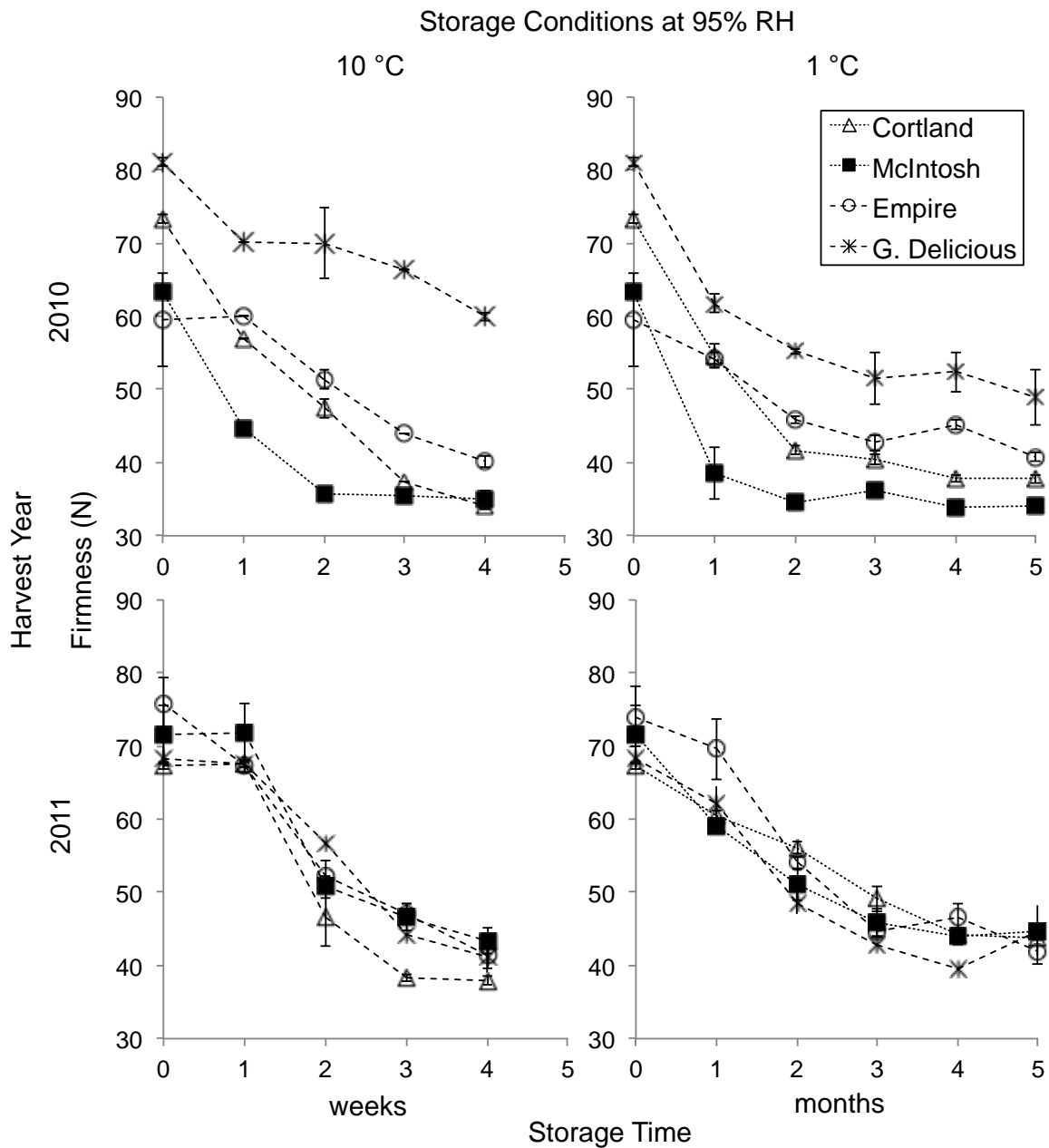


Figure 4.1 – Firmness of apples stored at 95% relative humidity (RH) at 10°C and 1°C for 4 weeks or 5 months, respectively, over 2 harvest years.

It is important to mention that for the 2010 harvest we also stored fruit at 21 °C for up to 20 days without RH control, in an attempt to verify the efficacy of accelerated fruit ripening practices more similar to those currently applied by manufacturers, but storage at those conditions was not able to yield comparable results between apples and

applesauce made from fruit stored at CS and accelerated fruit ripening storage. This is in agreement with previous reports by Louis and Massey (1989) that the lower the temperature employed for fruit tempering, the greater the reproducibility of changes occurring under CS, and that lack of controlled conditions is likely to yield variable and undesirable outcomes due to changes in the biochemistry of the apples which may induce breakdown with unexpected suddenness.

Applesauce Rheological Properties

USDA Consistency

Applesauce consistency is one of five attributes determining product grading along with flavor, color, absence of defects, and finish as defined by the USDA Grading Manual for Canned Applesauce (USDA, 2009). The flow of a grade A sauce shall not surpass 6.5 cm and any free-liquid present shall not have flow greater than 0.7 cm; the flow of a grade B sauce shall not surpass 8.5 cm and any free-liquid present shall not have flow greater than 1 cm; and sauce which fails to meet grade B requirements is of substandard consistency.

USDA consistency of sauces made from apples stored under CS (Figure 4.2) was variety-dependent showing harvest year and storage time effects ($p \leq 0.05$). Overall, it improved with progress of storage (thicker sauce with lower flow readings and minimal free-liquid changes) reaching optimum consistency between 2-3 months of fruit storage. Changes in consistency of sauce made from fruit stored at 10 °C were similar to that of

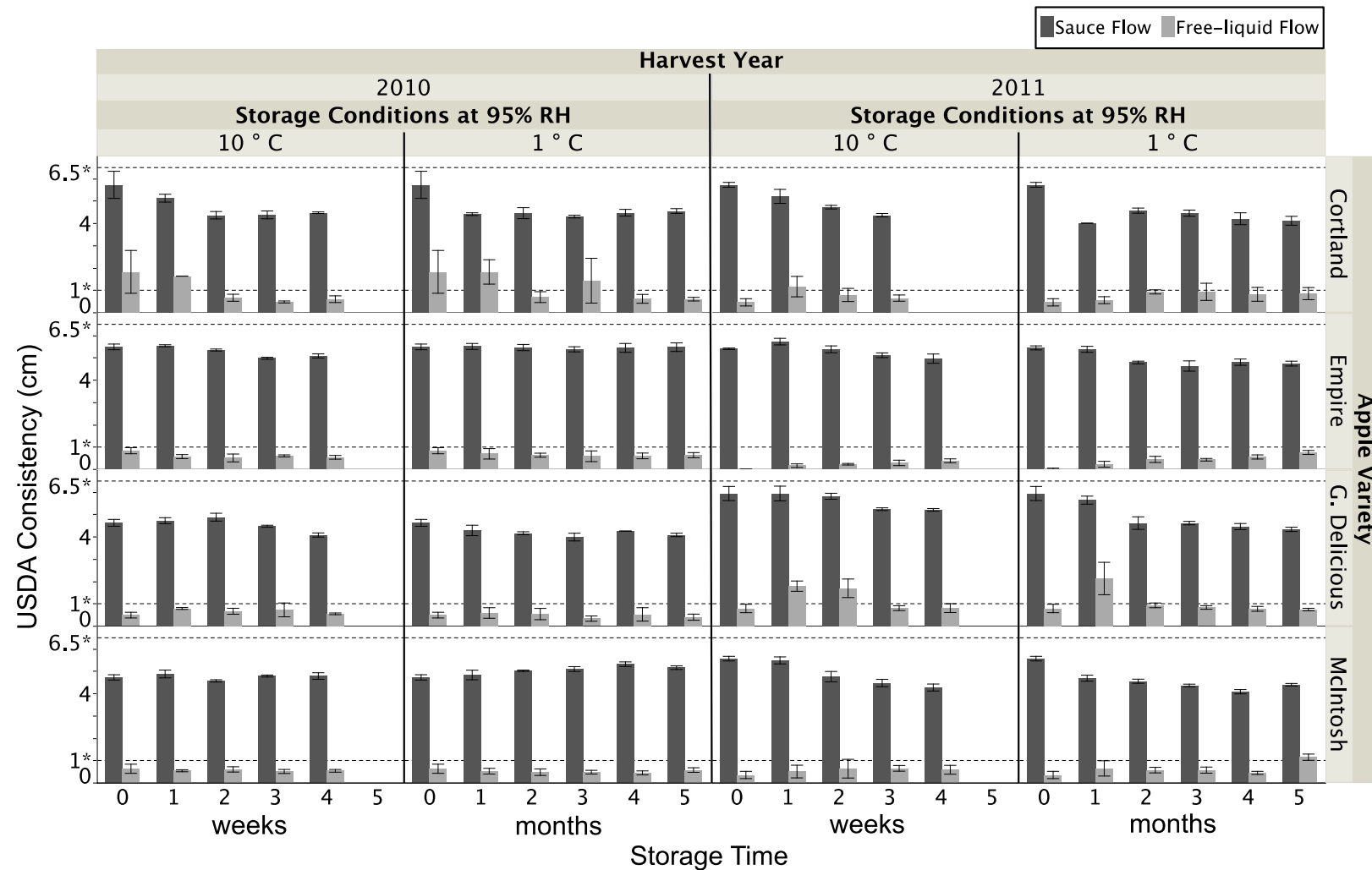


Figure 4.2 – USDA consistency (sauce and free-liquid flow) of applesauce made from apples stored at 95% relative humidity (RH) at 10 °C and at 1 °C for 4 weeks or 5 months, respectively, over 2 harvest years. *6.5 and 1 cm are tracking parameters for applesauce consistency grading.

CS fruit, but improvements occurred at a faster rate, with optimal consistency being reached between 2-3 weeks of fruit storage at 10 °C. Results suggest that manufacturers can effectively use the practice as a tool for improving rheological properties of sauce made from newly harvest fruit.

Our results are in agreement with previous studies by Wiley and Toldby (1960) on improvements of texture and overall grade of applesauce with fruit storage time. They differed, however, from those by Usiak and others (1995), who reported an increase in sauce flow in the beginning of fruit storage time under CS (4 months) followed by decrease towards the end of storage. In both cases, applesauce was obtained by blanching and magnitudes of consistency measurements differed greatly from our results. In a previous study performed by our group with hot-break applesauce, the fruit storage time effect on applesauce consistency was significantly reduced and similar trends and magnitudes to the literature were obtained (Chapter 2). Results were not representative of cold-break applesauce samples however, a processing method increasingly adopted by large-scale applesauce manufacturers in which applesauce is produced much more efficiently and cost-effectively.

Yield Stress and Consistency Index

Similar trends were observed for sauce yield stress and consistency index, rheological parameters of applesauce that denote applied stress required to initiate shear flow (Campanella and Pelleg, 1987) and the fluid's ability to resist motion when a shearing stress is applied (Barbosa-Canovas and others, 1996). Both parameters improved with fruit storage (higher readings due to stronger and more resistant structure

respectively) showing linear positive correlation ($R^2 = 0.95$), thus only consistency index is shown on Figure 4.3. Overall, most significant improvements in yield stress and consistency indexes were achieved after 2-3 weeks or months of fruit storage at 95% RH at 10 or 1 °C, respectively.

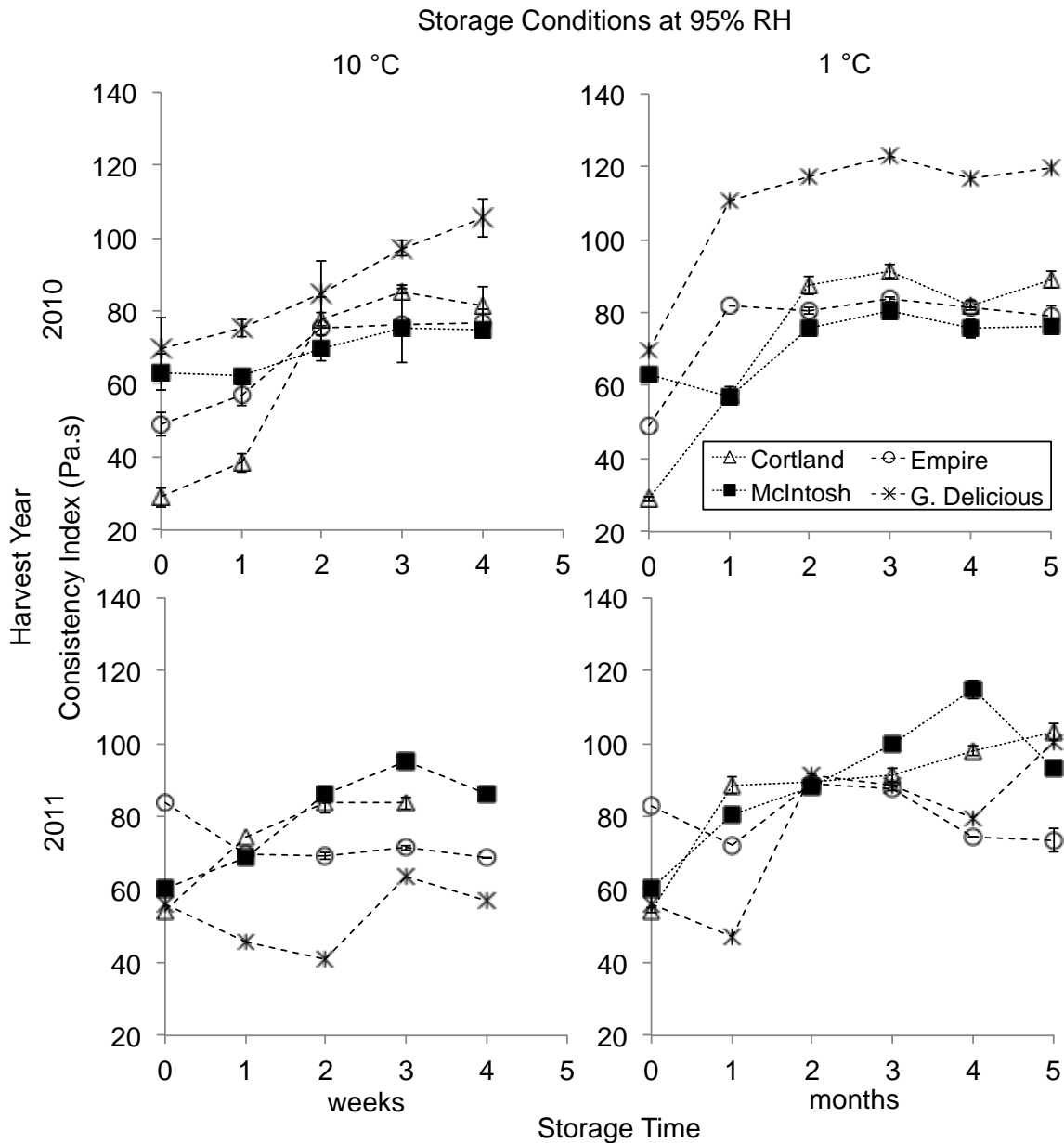


Figure 4.3 – Consistency Index of applesauce made from apples stored at 95% relative humidity (RH) at 10°C and 1°C for 4 weeks or 5 months, respectively, over 2 harvest years.

Our findings differ from those reported by Rao and others (1986) on the effect of fruit firmness on applesauce rheology. The authors observed a decrease in consistency index with lower fruit firmness (related to post-harvest fruit ripening) of Rhode Island Greening and Rome apples. Varietal differences as a source for the different behavior can be excluded as rheological parameters of the same varieties have also been studied by our group showing similar trends (Chapter 3); and, therefore, it could be related to the processing applied by the authors to obtain sauce, which included cooking fruit (hot-break), the use of significantly lower finisher rotational speeds (500, 700 and 900 rpm) and soluble solids adjustment to 16 °Brix for all samples to achieve a single Bostwick consistency value of 4.6 cm. In addition, in a previous study by our group with hot-break applesauce (Chapter 2) the effect of fruit post-harvest storage on sauce rheological properties was significantly reduced.

Regarding magnitudes of values, our observations – ranging 26.3 – 125.5 Pa.s for consistency index and 25.3 – 124.5 Pa for yield stress – were similar to those reported in the literature – 31–87 Pa and 7–50 Pa.s, respectively (Barbosa-Canovas and Peleg, 1983; Rao and others, 1986; Qiu and Rao, 1988; Shijvens and others, 1998), considering differences related to different methodologies to obtain sauce, rheological assessment methods and variations in raw material cultivar, ripeness and seasonal variations. In addition, as observed in previous studies (Chapter 3), yield stress and consistency index were negatively correlated to USDA consistency sauce flow ($R^2 \geq 0.53$) and could potentially be tracked by applesauce manufacturers for quality control purposes of rheological properties of applesauce.

Physical and Chemical Changes in Sauce Composition due to Fruit Ripening

Particle Size: Distribution (PSD), Mean (MPS) and Distribution Span (PSDS)

According to Mohr (1973) particle size in applesauce can be expressed as single values calculated to represent individual samples – commonly average diameter or mean particle size (MPS) –, but distribution pattern of sizes – or particle size distribution (PSD) – can be more informative in some cases. We further assessed particle size distribution span (PSDS), a single value that represents the width of the total particle size distribution.

Applesauce rheological properties were significantly affected by MPS. USDA consistency sauce flow was significantly lower and consistency index and yield stress significantly higher in sauce having lower MPS, which was dependent upon apple variety, harvest year and storage time (Figure 4.4). Most significant changes occurred after 3 weeks or months of fruit storage at 95% RH at 10 and 1 °C, during which time MPS significantly decreased (changing from 818 – 1002 μm at harvest to 597 – 892 μm after the mentioned storage time under either condition) and PSDS increased proportionally (changing from 0.88 – 1.53 at harvest to 1.23 – 2.04 μm after the suggested storage time), being overall stable after that – ranging 555 – 930 μm and 1.17 – 2.10, respectively.

Observed results and trends on sauce particle size over fruit storage were similar to those reported in the literature (Mohr, 1973 and 1989), and the effect of particle size on rheological properties of applesauce is in agreement with findings by Qiu and Rao (1988), who found negative correlation between average particle diameter and applesauce yield stress. PSDS was not a significant factor for sauce rheological properties. In a

previous study employing particularly challenging varieties for the achievement of optimal consistency products, however (Chapter 3), the parameter had a significant effect on USDA consistency sauce and free-liquid flow being higher for products showing lower sauce and free-liquid flow. It is possible that PSDS was not a significant factor in this particular study because overall varieties studied here produced sauce of optimal rheological properties with minimal liquid separation over fruit storage.

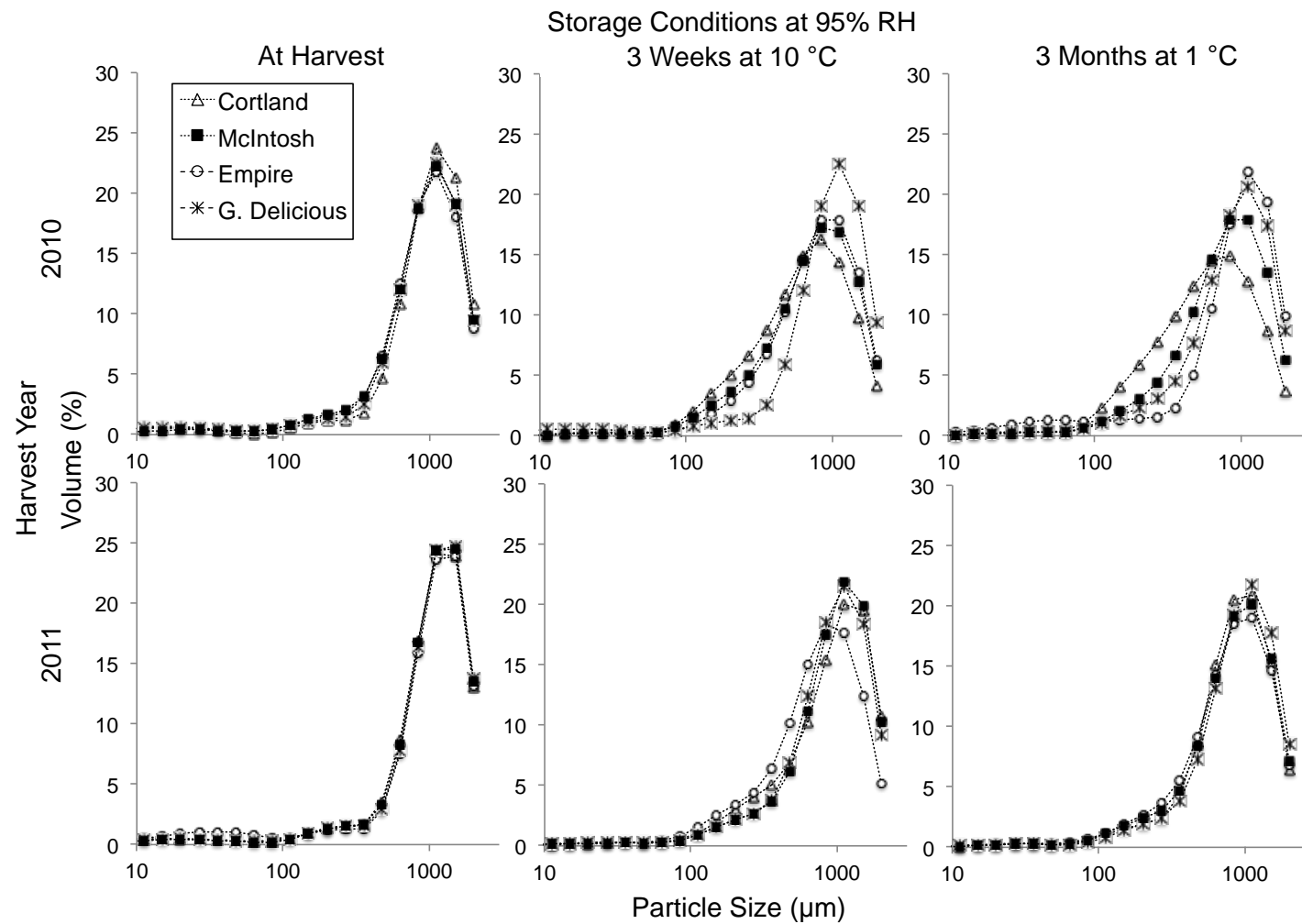


Figure 4.4 – Changes in particle size distribution of applesauce made from apples stored at 95% relative humidity (RH) at 10 °C and 1 °C for 3 weeks or 3 months, respectively, over 2 harvest years.

Moisture

Applesauce moisture ranged 83.7-90.5% across observations and was variety and harvest year dependent showing little variation over storage time and similar results over the two storage conditions studied. Among varieties and between harvest years, it was significantly different for Golden Delicious averaging 85.1 and 88.3% in 2010 and 2011 respectively but not significantly different between other varieties studied averaging 88.6 and 87% in 2010 and 2011, respectively. It was found to be a significant factor for free-liquid flow of sauce ($p \leq 0.05$), being higher for sauce showing more liquid separation, which is related to the effect of pulp content on sauce rheology (Beresovsky and others, 1995; Qiu and Rao, 1988). No other factor was significant for free-liquid flow with varieties in this study, which were overall optimal varieties for sauce consistency in both harvest years.

Starch

Investigation of starch in sauce made from newly harvested apples is important due to its thickening properties (Mason, 2009), which can affect sauce rheology. Starch degradation has long been used as a signal of apple maturity for harvest and traceable amounts in harvested fruit have been shown to quickly degrade into sugars (Smock and Neubert, 1950; Brookfield and others, 1997), and, therefore, differences in starch levels from year-to-year among varieties are likely related to apple maturity at harvest.

Starch did not have a significant effect on rheological properties of sauce in this particular study but general trends are in agreement with research expectations, being variety and harvest year dependent and quickly degrading over 2-3 weeks or months of

storage at 10 °C or 1 °C, respectively, after which time it became negligible ($\leq 0.01\%$), as previously observed (Chapter 3). Levels were higher in 2011 than in 2010 for applesauce made from freshly harvest apples – ranging 0.19 – 0.77 and 0.02 – 0.29% respectively –, being higher in sauce showing less USDA free-liquid flow. In 2011, average starch content in sauce in the beginning of fruit storage time was Empire (0.68%) \geq Cortland = McIntosh (0.46 and 0.43%) \geq Golden Delicious (0.24%). In 2010, starch levels were not significantly different among varieties, being $\leq 0.3\%$ in the beginning of the processing year (apple CS storage time = 0).

Calcium

Calcium is present in apples at a concentration of about 90 ppm (Pilgrim and others, 2011) and does not change with storage since minerals are not consumed during the life of the fruit (Smock and Neubert, 1950). Calcium assessment was carried out due to the possible influence of calcium concentration in gelling capability of pectin (Rees and others, 1982) present in applesauce.

Calcium did not significantly change over both storage conditions and was not significantly different across varieties studied and between harvest years averaging 25 and 29 ppm in 2010 and in 2011, respectively. Low levels of calcium concentration as compared to literature reports for apples, apple juice and pulp ranging 20–130 ppm (Perring, 1974; Nour and others, 2010) may be due to the assessment methodology, which was carried out in applesauce serum. Calcium concentration was not a significant factor for sauce rheological properties, indicating sufficient calcium is available in sauce to interact with pectins provided they have the suitable chemical structure.

Further Chemical Analysis of Sauce Made from CS Stored Fruit

Alcohol Insoluble Residue (AIR)

The residue after alcohol wash, AIR, is comprised of polysaccharides such as pectic substances and starch as well as small amounts of protein (Ladaniya, 2008). Its assessment was carried out due to their reported thickening and sedimentation prevention properties leading to reduction of liquid-separation in food matrixes (Stephen and Williams, 2006).

AIR of sauces (Table 4.1) did not significantly affect rheological properties of sauce made from varieties in this study, which showed minimal liquid separation and overall good AIR range ($\sim \geq 2.5\%$).

AIR was dependent on apple variety, harvest year, fruit storage time and their interaction ($p \leq 0.05$), ranging 1.67-3.76% and changing according to changes in starch and total soluble pectin (TSP), both observed to be important factors for prevention of free-liquid flow in applesauce in previous studies (Chapters 2 and 3). AIR quantification is much simpler and involves fewer investments in equipment than that of starch and pectin and could potentially be a more effective tracking parameter for product quality control purposes of the polysaccharides affecting sauce rheological properties by sauce manufacturers. Range of results observed is in agreement with previous reports on AIR content in apples and applesauce – 1.76-5.48% (De Vries, 1981; Fischer and others, 1994; Colin-Henrion, 2009).

Total Soluble Pectin (TSP) and Pectin Degree of Methoxylation (PDM)

Pectins are a group of polysaccharides having d-galacturonic acid as the principal sub-unit joined by α -(1-4). An important factor characterizing pectin chains is its degree of esterification with methyl alcohol or degree of methoxylation (DM), which signals tendency for cross-linking with calcium ions as the DM of pectin decreases (Van Buren, 1991). Importance of pectin content to applesauce rheological properties has been previously reported in the literature (Toldby and Wiley, 1962; Rao and others, 1986) while importance of its chemical structure has been suggested (La Belle, 1981; Usiak and others, 1995).

Both TSP and PDM of sauces (Table 4.1) were significantly affected by apple variety, harvest year and storage time ($p \leq 0.05$) and were significant factors for rheological properties of sauce averaging 0.19-0.42% and 32.6-94.7%, respectively. TSP was significantly higher in sauce having higher yield stress ($p \leq 0.01$) while PDM was significantly lower in sauce having higher consistency index ($p \leq 0.05$). These results are in support of the role of pectin content and chemical structure for rheological properties of cold-break applesauce observed in a previous study (Chapter 3). This information is of interest for applesauce manufacturers for selection of processing varieties, which should be preferably higher in TSP content (or AIR due to simpler quantification) and lower PDM for the achievement of products of optimal rheological properties according to this study.

General storage time effects observed on TSP and PDM might be explained by de-polymerization of pectin fractions (insoluble to soluble) and de-esterification of the uronide carboxyl groups by enzymatic activity notably of polygalacturonase and pectin

methylesterase associated with fruit ripening (Knee and Bartley, 1981; Fischer, 1991; Van Burren, 1991); while high standard deviations in some PDM results, in particular, might have been due to cumulative errors in the extraction methodology of samples leading to considerable sample-to-sample variability and unexpected outcomes. Finally, harvest year effects in TSP could be related to weather conditions affecting fruit composition (Smock and Neubert, 1950).

Toldby and Wiley (1962) previously reported the effect of pectin content in sauce liquid-solid separation observing a higher range of results (0.55 – 0.78%) probably due to use of different assessment methods and methodologies for obtaining sauce other than inherent differences in horticultural products related to variety, maturity at harvest, ripening stage and harvest season. Other sources report pectin content in apples (at harvest and with progress of storage) and in applesauce to range 0.17 – 0.55% (McClendon and others, 1959; De Vries, 1981; Lo Scalzo and others, 2005; Vanoli and others, 2009; Le Bourvellec and others, 2011).

The effect of lower PDM in food systems has been studied more extensively for tomatoes being reported to significantly improve firmness of diced products and to cause adverse effects in the consistency of juice (McColloch and Kertsz, 1949; Castaldo and others, 1995; Grassin and others, 2002; Anthon and others, 2005; Anthon and Barret, 2010). Pressey and Avants (1982) explained that, in fluid tomato products, pectin methyl-esterase (PME) catalyzed pectin de-esterification might cause pectin to precipitate, increasing the tendency of juice serum to separate from juice solids. Applesauce is a dispersion of solids in a liquid phase where very little sedimentation occurs (Rao, 1977); while fluid tomato products are best described as suspensions, where considerable

sedimentation can occur, and therefore, the potential difference in the effect of lower PDM between the food matrixes. Our study findings support the hypothesis that, in applesauce, higher calcium cross-links by pectins having lower PDM have a positive effect on applesauce consistency, as observed in previous studies (Chapter 3).

Results and trends for PDM are overall comparable to literature on apple varieties at harvest and with progress of storage time and varietal applesauce ranging 47 – 88% (De Vries and others, 1984; Klein and others, 1995; Johnston and others, 2002; Anthon and Barret, 2008; Rascón-Chu and others, 2009; Le Bourvellec and others, 2011).

Table 4.1. Chemical parameters affecting applesauce rheological properties: alcohol insoluble residue (AIR); total soluble pectin (TSP) and pectin degree of methylation (PDM) of sauce made from apples stored at 1 °C and 95% relative humidity (RH) for 5 months, over 2 harvest years. Different letters indicate differences over storage time for a given variety within a harvest year.

Parameter	Apple Variety	Harvest Year					
		2010			2011		
		Storage Conditions: 1 °C at 95% RH					
		Storage Time (months)			Storage Time (months)		
		0	3	5	0	3	5
AIR (%)	Cortland	2.94 ± 0.7 ^a	2.49 ± 0.07 ^b	2.69 ± 0.07 ^b	3.01 ± 0.01 ^a	2.53 ± 0.03 ^a	2.89 ± 0.74 ^a
	Empire	2.62 ± 0.5 ^a	2.53 ± 0.23 ^a	2.13 ± 0.06 ^a	3.73 ± 0.04 ^a	3.02 ± 0.42 ^b	2.37 ± 0.13 ^c
	G. Delicious	2.96 ± 0.10 ^a	2.36 ± 0.35 ^b	2.94 ± 0.37 ^{ab}	2.39 ± 0.06 ^a	2.19 ± 0.14 ^{ab}	2.02 ± 0.12 ^b
	McIntosh	2.07 ± 0.09 ^a	2.13 ± 0.16 ^a	1.89 ± 0.25 ^a	2.63 ± 0.16 ^a	2.59 ± 0.03 ^a	2.08 ± 0.08 ^b
TSP (%)	Cortland	0.28 ± 0.01 ^c	0.40 ± 0.01 ^b	0.43 ± 0.01 ^a	0.23 ± 0.03 ^b	0.28 ± 0.03 ^{ab}	0.31 ± 0.03 ^a
	Empire	0.23 ± 0.03 ^b	0.32 ± 0.04 ^a	0.26 ± 0.05 ^{ab}	0.27 ± 0.05 ^{ab}	0.33 ± 0.03 ^a	0.20 ± 0.01 ^b
	G. Delicious	0.19 ± 0.01 ^b	0.23 ± 0.03 ^b	0.33 ± 0.05 ^a	0.32 ± 0.01 ^a	0.24 ± 0.02 ^b	0.25 ± 0.03 ^b
	McIntosh	0.21 ± 0.03 ^c	0.34 ± 0.01 ^a	0.28 ± 0.02 ^b	0.21 ± 0.01 ^b	0.25 ± 0.01 ^a	0.19 ± 0.01 ^c
PDM (%)	Cortland	87.9 ± 31.7 ^a	40.2 ± 2.8 ^b	32.6 ± 2.2 ^b	60.5 ± 6.5 ^a	60.5 ± 14.7 ^a	64.7 ± 12.9 ^a
	Empire	44.6 ± 8.23 ^a	42.8 ± 8.5 ^a	48.2 ± 9.6 ^a	72.8 ± 3.7 ^a	68.9 ± 2.9 ^a	67.1 ± 4.2 ^a
	G. Delicious	54.8 ± 0.8 ^a	55.5 ± 3.6 ^a	47.8 ± 8.0 ^a	48.5 ± 2.2 ^b	54.5 ± 3.7 ^a	44.2 ± 0.8 ^b
	McIntosh	66.1 ± 3.13 ^a	69.7 ± 3.8 ^a	69.7 ± 0.57 ^a	66.8 ± 1.2 ^c	94.7 ± 7.4 ^a	84.4 ± 1.6 ^b

Conclusions

Accelerated post-harvest fruit ripening seems to be a beneficial practice to improve the rheological properties of applesauce made from freshly harvested apples, but the practice must be carried out under controlled conditions for optimal results – 10 °C at 95% RH for 2-3 weeks is suggested. Rheological improvements of sauce over fruit storage time were correlated to MPS reduction while general differences among varieties and harvest years were related to differences in chemical composition of sauce (TSP and PDM, which were higher and lower, respectively, in sauce having higher yield stress and consistency index). AIR content in applesauce was dependent upon TSP and starch. Starch quickly degraded over fruit storage time thus manufacturers could track AIR for quality control purposes of soluble pectin content in finished products. Finally, during application of accelerated post-harvest ripening practices, fruit ripening indicators such as firmness, TA, pH and soluble solids can potentially help determining best time for processing specific varieties into sauce.

References

- Ahmed A, Labavitch, J. 1977. A simplified method for accurate determination of cell wall uronide content. *J Food Biochem* 1:361–365.
- Anthon, G. E., & Barrett, D. M. 2004. Comparison of three colorimetric reagents for the determination of methanol with alcohol oxidase. Application to the assay of pectin methylesterase. *J Agric Food Chem* 52: 3749–3753.
- Anthon GE, Blot L, Barrett DM. 2005. Improved firmness in calcified diced tomatoes by temperature activation of pectin methylesterase. *J Food Sci* 70:342-347.
- Anthon GE, Barrett DM. 2008. Combined enzymatic and colorimetric method for determining the uronic acid and methylester content of pectin: Application to tomato products. *Food Chem* 110(1):239-47.

- Anthon GE, Barrett DM. 2010. Changes in pectin methylesterification and accumulation of methanol during production of diced tomatoes. *J.Food Eng.* 97(3):367-72.
- AOAC International. 2000. Official Methods of Analysis of AOAC International. 17th ed. Arlington: AOAC International. 2200 p.
- Barbosa-Cánovas GV, Peleg M. 1983. Flow parameters of selected commercial semi-liquid food products. *J Texture Stud* 14(3):213-34.
- Barbosa-Cánovas GV, Kokini JL, Ma L, Ibarz A. 1996. The rheology of semiliquid foods. *Adv Food Nutr Res* 39:1-69.
- Beresovsky N, Kopelman IJ, Mizrahi S. 1995. The role of pulp interparticle interaction in determining tomato juice viscosity. *J Food Process Preserv* 19(2):133-46.
- Blumenkrantz, N., & Asboe-Hansen, G. 1973. New method for quantitative determination of uronic acids. *Anal Biochem* 54: 484–489.
- Brookfield P, Murphy P, Harker R, MacRae E. 1997. Starch degradation and starch pattern indices; interpretation and relationship to maturity. *Postharvest Biol.Technol* 11(1):23-30.
- Calvin L, Martin P. 2011. The U. S. Produce Industry and Labor: Facing the Future in a Global Economy, ERR-106, U.S. Department of Agriculture, Economic Research Service, November 2010. DIANE Publishing. 57p.
- Campanella OH, Peleg M. 1987. Determination of the yield stress of semi-liquid foods from squeezing flow data. *J Food Sci* 52(1):214-5.
- Castaldo D, Servillo L, Laratta B, Fasanaro G, Villari G, De Giorgi A, Giovane A. 1995. Preparation of high-consistency vegetable products: tomato pulps (part II). *Industrial Conserve* 70:253–258.
- Chin, L. H., Ali, Z. M., & Lazan, H. 1999. Cell wall modifications, degrading enzymes and softening of carambola fruit during ripening. *J Exp Bot* 50(335): 767–775.
- Colin-Henrion M, Mehinagic E, Renard CMGC, Richomme P, Jourjon F. 2009. From apple to applesauce: Processing effects on dietary fibres and cell wall polysaccharides. *Food Chem* 117(2):254-60.
- De Vries JA, Voragen AGJ, Rombouts FM, Pilnik W. 1981. Extraction and purification of pectins from Alcohol Insoluble Solids from ripe and unripe apples. *Carbohydr.Polym* 1(2):117-27.
- De Vries JA, Rombouts FM, Voragen AGJ, Pilnik W. 1984. Comparison of the structural features of apple and citrus pectic substances. *Carbohydr.Polym* 4(2):89-101.

- FDA: 21CFR145.110 – Canned Applesauce [Internet]. Silver Spring, MD: U.S. Food and Drug Administration [Accessed 2012 Sep 17]. Available from: <http://www.accessdata.fda.gov/scripts/cdrh/cfdocs/cfcfr/CFRSearch.cfm?fr=145.110>.
- Fischer RL, Bennett A. 1991. Role of cell wall hydrolases in fruit ripening. *Annual review of plant biology* 42(1):675-703.
- Fischer M, Arrigoni E, Amadò R. 1994. Changes in the pectic substances of apples during development and postharvest ripening. Part 2: Analysis of the pectic fractions. *Carbohydr.Polym* 25(3):167-75.
- Grassin C. 2002. Firm up your fruit! *Fruit Processing* 12:208–211.
- Johnston J, Hewett E, Hertog M. 2002. Postharvest softening of apple (*Malus domestica*) fruit: A review. *N Z J Crop Hortic Sci* 30(3):145-60.
- Klein JD, Hanzon J, Irwin PL, Shalom NB, Luria S. 1995. Pectin esterase activity and pectin methyl esterification in heated golden delicious apples. *Phytochemistry* 39(3):491-494.
- Knee M, Bartley IM. 1981. Composition and metabolism of cell-wall polysaccharides in ripening fruit. In: *Recent Advances in Biochemistry of Fruits and Vegetables*. New York: Academic Press. P 133-148.
- La Belle RL. 1981. Apple quality characteristics as related to various processed products. In: R. Teranishi and H. Barrera-Benitez. *Quality of selected fruits and vegetables of North America*. ACS Symposium Series. Washington: American Chemical Society. p. 61–76.
- Ladaniya MS. 2008. *Citrus fruit: biology, technology and evaluation*. San Diego: Academic Press. 558p.
- Le Bourvellec C, Bouzerzour K, Ginies C, Regis S, Plé Y, Renard CMGC. 2011. Phenolic and polysaccharidic composition of applesauce is close to that of apple flesh. *Journal of Food Composition and Analysis* 24(4–5):537-47.
- Lo Scalzo R, Forni E, Lupi D, Giudetti G, Testoni A. 2005. Changes of pectic composition of ‘annurca’ apple fruit after storage. *Food Chem* 93(3):521-530.
- Louis M, Massey JR. 1989. Harvesting, Storing and Handling Processing Apples. In: Downing DL. *Processed apple products*. New York: Van Nostrand Reinhold. P 215-238.

- Mason WR. 2009. Starch use in foods. In: BeMiller J, Whistler R. Starch Chemistry and Technology. 3rd edn. San Diego: Academic Press. p 745-95.
- Massey Jr. LM. 1989. Harvesting, storing and handling processing apples. In: Downing DL. Processed apple products. New York: Van Nostrand Reinhold. P 31-51.
- McClendon JH, Woodmansee CW, Somers GF. 1959. On the occurrence of free-galacturonic acid in apples and tomatoes. *Plant Physiol* 34(4):389-91.
- McLellan MR, Massey LM. 1984. Effect of postharvest storage and ripening of apples on the sensory quality of processed applesauce. *J. Food Sci* 49(5):1323-6.
- McColloch RJ, Kertesz ZI. 1949. *Food Tech* 3: 923.
- Mohr WP. 1973. Applesauce grain. *J Texture Stud* 4(2):263-8.
- Mohr WP. 1989. Influence of cultivar, fruit maturity and fruit anatomy on apple sauce particle size and texture. *Int J Food Sci Tech* 24(4):403-13.
- Nour V, Trandafir I, Ionica ME. 2010. Compositional characteristics of fruits of several apple (*Malus domestica* Borkh.) cultivars. *Not Bot Hort Agrobot Cluj* 38 (3): 228-233.
- Perring MA. 1974. The chemical composition of apples. XI. An extraction technique suitable for the rapid determination of calcium, but not potassium and magnesium, in the fruit. *J Sci Food Agric* 25(3): 237–245.
- Pilgrim GW, Walter RH, Oakenfull DG. 1991. Jams, jellies and preserves. In: The chemistry and technology of pectin. San Diego: Academic Press. P 23–50.
- Pressey R, Avants, J. 1982. Solubilization of cell walls by tomato polygalacturonase: effects of pectinesterases. *J Food Biochem* 6: 57–74.
- Qiu C, Rao MA. 1988. Role of pulp content and particle size in yield stress of apple sauce. *J Food Sci* 53(4):1165-1170.
- Rao MA. 1977. Rheology of liquid foods – a review. *J Texture Stud* 8:135.
- Rao MA, Cooley HJ, Nogueira JN, McLellan MR. 1986. Rheology of apple sauce: effect of apple cultivar, firmness, and processing parameters. *J Food Sci* 51(1):176-179.
- Rascón-Chu A, Martínez-López AL, Carvajal-Millán E, Ponce de León-Renova NE, Márquez-Escalante JA, Romo-Chacón A. 2009. Pectin from low quality ‘golden delicious’ apples: composition and gelling capability. *Food Chem* 116(1):101-103.

- Rees DA, Morris ER, Thorn D, Madden JK. 1982. Shapes and interactions of carbohydrate chains. In: Aspinall GO. The Polysaccharides. New York: Academic Press. P 195–290.
- Schijvens EPHM, Van Vliet T, Van Dijk C. 1998. Effect of Processing Conditions on the composition and rheological properties of applesauce. *J Texture Stud* 29(2):123-143.
- Sila, D. N., Doungra, E., Smout, C., Van Loey, A., & Hendrickx, M. (2006). Pectin fraction interconversions: Insight into understanding texture evolution of thermally processed carrots. *Journal of Agricultural and Food Chemistry*, 54(22), 8471–8479.
- Skog LJ, Chu CL. 2003. APPLES. In: Caballero B. Encyclopedia of Food Sciences and Nutrition (Second Edition). Oxford: Academic Press. p 290-4.
- Smock RM, Neubert AM. 1950. Apples and apple products. New York: Interscience Publishers. P 486.
- Stephen AM, Glyn OP, Williams PA. 2006. Food polysaccharides and their applications. New York: CRC Press. 752 p.
- Toldby V, Willey R. 1962. Liquid-solids separation, a problem in processed applesauce. *J Am Soc Hortic Sci* 81:78-90.
- U.S. Apple Association: Production and Utilization Analysis 2011 [Internet]. Vienna, VA: U.S. Apple Association. [Accessed 2013 March 8]. Available from: <http://www.yvgsa.com/pdf/facts/USApple2011ProductionAnalysis.pdf>.
- USDA: Grading Manual for Canned Applesauce [Internet]. Washington, D.C.: United States Department of Agriculture [Accessed 2009 Sep 19]. Available from: <http://www.usda.gov>.
- Usiak AMG, Bourne MC, Rao MA. 1995. Blanch temperature/time effects on rheological properties of applesauce. *J.Food Sci.* 60(6):1289-1291.
- Van Buren JP. 1991. Function of pectin in plant tissue structure and firmness. In: Walter RH. The chemistry and technology of pectin. San Diego: Academic Press. p 1-22.
- Vanoli M, Zerbini PE, Spinelli L, Torricelli A, Rizzolo A. 2009. Polyuronide content and correlation to optical properties measured by time-resolved reflectance spectroscopy in ‘Jonagored’ apples stored in normal and controlled atmosphere. *Food Chem* 115(4):1450-1457.
- Wiley RC, Toldby V. 1960. Factors affecting the quality of canned applesauce. *Proc Am Soc Hortic Sci* 76: 112–23.

CHAPTER 5:
CONSISTENCY OF COLD-BREAK APPLESAUCE MADE FROM
CONTROLLED ATMOSPHERE STORED APPLES (*Malus domestica* Borkh.)

ABSTRACT: Applesauce manufacturers have been facing challenges to achieve products of optimal consistency when processing freshly harvested and controlled atmosphere (CA) stored fruit. In 2010 and 2011, four different apple varieties (Crispin, Idared, Jonagold and Rome Beauty) were stored up to 5 weeks at 10 °C and 95% relative humidity (RH) immediately after harvest – control group – and after CA storage – 1-4 °C, 95-98% RH, 1-3% O₂ and 1-4% CO₂ for 7-10 months – CAS group. Applesauce was made weekly from both groups following a cold-break procedure to assess and compare sauce rheological properties over fruit storage time. Apples were evaluated for ripening indicators (firmness, pH, acidity, soluble solids) and single-variety sauce was tested for rheology (USDA consistency, consistency index, yield stress); particle size distribution, mean particle size (MPS) and particle size distribution span (PSDS); pH; acidity; soluble solids; moisture; calcium and starch. Results were analyzed by ANOVA and significant differences among means determined by Tukey's test ($p \leq 0.05$). Overall, rheological properties of sauce made from CAS apples were different than those of sauce made from freshly-harvested fruit ($p \leq 0.05$). Firmness readings indicate significant fruit softening over storage at 10 °C and 95% RH of fresh, but not for most CAS apples, which underwent changes over the CA storage period. Similar behavior was found for particle size distribution and starch content, significant factors for rheological properties of sauce. Further chemical analysis on applesauce made from fresh and CAS apples – alcohol insoluble residue (AIR); total soluble pectin (TSP) and pectin degree of methylation (PDM) – indicate that those parameters can also change significantly over CA storage affecting varietal processing performance into sauce.

Keywords: applesauce, controlled atmosphere storage, consistency, rheology.

Practical Application: Controlled atmosphere (CA) storage of apples allows availability of fruit for applesauce processing throughout the year, yet little is known about how the storage practice affects processed apple products. Cold-break applesauce manufacturers face challenges in achieving products of desirable consistency when processing newly harvested apples and fruit coming out of CA storage. In previous studies, our group has demonstrated that rheological properties of sauce are significantly improved by post-harvest fruit ripening. Information about how rheological properties and physical and chemical composition of applesauce made with apples coming out of CA storage compare to sauce made from fresh and post-harvest ripened fruit, can potentially assist manufacturers with raw material storage and blend management targeting product optimization.

Introduction

Controlled atmosphere (CA) storage is considered the most effective means of prolonged storage for most processing apple cultivars. It consists of holding fruit in a gas-tight refrigerated room, which can extend the usable life of apples 7-9 months or more depending on cultivar and particular product being manufactured (Louis and Massey, 1989). The storage atmosphere is typically composed of reduced levels of oxygen and increased levels of carbon dioxide at low temperature – 1-3% O₂; 1-5% CO₂ and 0-4 °C, respectively –, three principles applied in combination to reduce ethylene synthesis and the respiratory rate of the fruit dramatically along with associated ripening changes (Kader, 1986; Watkins, 2003).

Although CA storage of apples has been studied since the 20's (Kidd and West, 1927) and commercial application has been reported since the 50's (Smock and Neubert, 1950), available studies on the subject are heavily focused on firmness of table quality fruit (McLellan and others, 1990; Siddiqui and others, 1996; Gwanpua and others, 2012), while there are few reports on the impact of using CAS apples on processed products (Lidster and others, 1984; Massey and McLellan, 1985; Rocha and De Moraes, 2000).

In the case of applesauce, a distinctively American product prepared from comminuted or chopped apples (FDA 2012), CA storage is employed by manufacturers in addition to regular atmosphere cold storage (CS) – 0-4 °C and 95-98% relative humidity (RH) (Patchen, 1971) – for guaranteeing availability of processing quality fruit year-round as the harvest season of most processing cultivars lasts between August and October in the U.S (Calvin and Martin, 2011). The Department of Agriculture reports apple holdings in cold storage (including CA) as long as 11 months (USDA, 2012).

For cold-break applesauce manufacturers, processing challenges related to product rheological properties such as thin sauce and substandard consistency are typically faced when CA stored apple stocks start being used, as those under CS are consumed or reach the end of storage life – between 5-7 months after harvest based on variety; and only CA stored apples are available for processing prior to the new harvest season. Similar challenges are reported when newly-harvested apples are used, and, as a result, CA stored fruit is believed to have processing performance similar to that of fresh fruit although CA storage is reported to affect table quality fruit, only not as much as other storage practices (Vanoli and others, 2009).

Drake and others (1979) studied the influence of CA storage on the quality of applesauce from Golden Delicious apples and reported that no effect was observed on the consistency of sauce when compared to sauce made from CS apples. The methodology to obtain sauce followed by the authors involved a hot-break-process of steam-cooked apple dice, and cooking time was a significant factor for sauce consistency. Many applesauce-processing facilities have evolved from a hot-break process to a more efficient cold-break one, resulting in challenges to meet the targeted rheological properties of products. In a previous study by our group, when the hot-break method was applied to obtain sauce from CS apples, the fruit storage time effect on applesauce rheological parameters was significantly reduced (Chapter 2), but rheological parameters of cooked sauce were significantly different than those of commercially available products, thus, cold-break processing was utilized to meet current practices followed by large manufacturers.

Applesauce rheological parameters are reported to be dependent on apple firmness and applesauce particle size, starch, pectin and pulp content (Toldby and Wiley, 1962; Rao and others, 1986; Qiu and Rao, 1988; Usiak and others, 1995), which can be influenced by fruit ripeness (La Belle, 1981; Mohr, 1973 and 1989) and therefore storage practice and time.

Our objective was to study these factors on CA stored apples and resulting varietal sauce in relation to product rheological properties; and to establish comparisons with freshly harvested apples and their resulting sauce; as means to provide valuable information regarding the use of CA stored apples for cold-break applesauce processing.

Materials and Methods

Apples

Apples (*Malus domestica* Borkh.), which included Crispin, Idared, Jonagold and Rome Beauty were harvested in October of 2010 and 2011 from apple farms located in New York State and were freshly delivered to processing pilot plant at Cornell University (control group); or were stored under controlled atmosphere (CA) storage – 1-4 °C, 95-98% RH, 1-3% O₂ and 1-4% CO₂ – for 7-10 months (Crispin = 7 months in 2010 and all other apples = 10 months at both harvest years) after which time they were also delivered to the processing pilot plant (CAS group). Both groups were stored at 10 °C and 95 % relative humidity for up to 5 weeks, until processed into applesauce, carried out weekly.

Apple Maturity Indicators and Applesauce Processing

Prior to processing, apples were weighed and tested for firmness using a hand-held penetrometer model FT 327 (Wagner Instruments, Greenwich, CT). Sauce making followed industrial practices according to information provided by applesauce manufacturers: apples (~ 15 kg) were fed through turbo extractor (1.6 mm screen, 8 mm gap, 1800 rpm; Bertocchi CX5, Bertocchi SLR., Parma, Italy) and 15% water (w/w) was added to the sauce (to simulate water pick-up by direct steam injection) which was then heated in steam kettle at 96-98 °C for 6 min and hot-filled to 8 oz glass jars. Jars were inverted for 3 min for cap sterilization, cooled in water bath and stored at 1 °C until analysis. A sample of comminuted apples (turbo extractor output) was collected and pressed through cheesecloth to obtain juice which was tested for pH using a bench-top

Thermo Scientific pHmeter model Orion 3-Star (Cellomics, Pittsburgh, PA); titratable acidity (TA) – through titration with NaOH 0.1 N and recorded as % malic acid; and soluble solids – according to AOAC (2000) utilizing a bench-top refractometer model Leica Auto Abbe (Leica Inc., Buffalo, NY).

Applesauce Analysis

Applesauce yield stress and consistency index of samples were determined using a vane spindle model V-73 in a Brookfield DV-III Ultra programmable rheometer at constant temperature (25 °C) with software package RheoCalc (equipment and program from Brookfield Engineering Laboratories, INC. Middleboro, MA). Yield stress and consistency index (K) were calculated using the Casson and power law models, respectively, from shear-rate and shear-stress data obtained by subjecting samples to 0.5 s⁻¹ increments of shear-rate from 0.5 to 3.0 s⁻¹ upward and backward with 1 min hold at each shear-rate prior to data collection every 1-min, during a total time of 11 min. USDA consistency was measured according to the Grading Manual for Canned Applesauce (USDA, 2009) and qualitative consistency grading was assigned. The volume-based particle size distribution (PSD) Mean particle size (MPS) and particle size distribution span (PSDS) were assessed using a Malvern laser diffraction unit model Mastersizer 2000 (Malvern Instruments Inc., Westborough, MA). MPS was calculated as the volume-based mean particle diameter ($\sum_i n_i d_i^4 / \sum_i n_i d_i^3$ where n_i is the number of particles of diameter d_i) and PSDS was calculated as width of the volume-based particle size distribution ($(d_{90th\ percentile} - d_{10th\ percentile}) / d_{50th\ percentile}$). Applesauce moisture was obtained according to AOAC (2000). Applesauce pH was obtained as previously

described for apple slices. Applesauce samples were centrifuged at 17000 rpm for 30 min and the supernatant (applesauce serum) was collected and stored at -10 °C until further analysis. Alcohol insoluble residue (AIR), total soluble pectin (TSP) and pectin degree of methoxylation (PDM) analysis were carried out as previously described in detail in Chapter 2. Isolation of AIR was carried out according to Mcfeeters & Armstrong (1984) and reported as % AIR in applesauce. AIR was ground and pulverized for extraction of water- and cheletor-soluble pectin fractions (WSP and CSP, respectively). The WSP and CSP fractions were obtained following the procedures by Sila and others (2006) and Chin and others (1999), respectively. Each fraction was analyzed for Galacturonic acid (GalA) and methanol for determining pectin content (as GalA equivalent) and pectin degree of methoxylation (as the ratio of the molar amount of methanol esters to the molar amount of galacturonic acid residues). The GalA content in pectin fractions was determined by hydrolysis in H₂SO₄/ tetraborate solution (0.0125 M solution of sodium tetraborate in concentrated sulfuric acid) as described by Ahmed and Labavitch (1977) with subsequent colorimetric determination according to Blumenkrantz and Asboe-Hansen (1973) by using a Barnstead Turner SP830 Spectrophotometer (Barnstead International, Dubuque, IA). The methanol concentration was determined by alkaline hydrolysis of 1 volume of sample in 2 volumes of 0.5 M NaOH and subsequent incubation at room temperature for 1 hour followed by neutralization with 1 volume of 1 M HCl according to Anthon and Barrett (2008). The amount of methanol was determined using alcohol oxidase and Purpald as described by Anthon and Barrett (2004). WSP and CSP were proportionally combined as fractions of applesauce AIR in order to obtain values for TSP and PDM of sauces. Additionally, applesauce samples were centrifuged at 17000 rpm for 30 min and

the supernatant (applesauce serum) was collected through filtration also using Whatman filter paper 55 mm. The serum was stored at -10 °C until further analysis. Applesauce serum titratable acidity was obtained as previously described for apples. Calcium concentration in applesauce serum was determined using Calcium-Arsenazo quantification kit (BEN Biochemical Enterprise, Milano, Italy). Starch analysis was performed through iodine-iodide 0.01 N reaction measuring absorbance at 570 nm referring to a standard curve of known starch concentrations in a Turner spectrophotometer model Barnstead SP-830 (Turner Biosystems, Dubuque, IA) reported as % starch (g/100 ml).

Statistical Analysis

Two batches of apples were processed into applesauce generating two replicates, resulting in a total of 4 samples for each experimental point. Measurement for all experimental units was conducted in duplicate or triplicate and results were expressed as means and standard deviations. Data was analyzed by ANOVA and significant differences among means adopting a 95 % confidence interval ($p \leq 0.05$) were determined by Tukey's test using JMP® 9.0 statistical software (SAS institute Inc., Cary, NC).

Results and Discussion

Apple Ripening Indicators

According to Wiley and Binkley (1989), rather simple and quick tests have been used to check ripeness of apples by applesauce processors such as pressure test (firmness); titratable acidity (TA) and soluble solids (SS) although attempts to relate them to final product quality have been unsuccessful due to overall quality dependency on other aspects such as processing and final product formulation. We additionally tracked these parameters to assess the extent of fruit ripening occurring to apples under extended CA storage and its relationship with changes in physical and chemical parameters of sauce affecting its rheological properties.

Firmness of apples (Figure 5.1) immediately out of CA was significantly lower than that of freshly harvested fruit ($p \leq 0.05$) and was not significantly different over storage at 10 °C and 95% RH for 5 weeks following the CA storage period – CAS group – for most varieties; being similar to levels observed for fresh apples at the end of storage at 10 °C and 95% RH for 4 weeks – FS group. TA and pH were also significantly different, changing for both FS and CAS (ranging 0.66-0.38% and 3.3-3.6; and 0.50-0.18% and 3.5-4.1; respectively, accounting for both harvest years); while small differences in SS were variety and harvest year dependent, with overall stable levels ranging 8.8 – 14.6 °Brix across the study. These parameters signal that significant ripening changes do occur to apples stored under CA although the practice extends fruit storage life significantly.

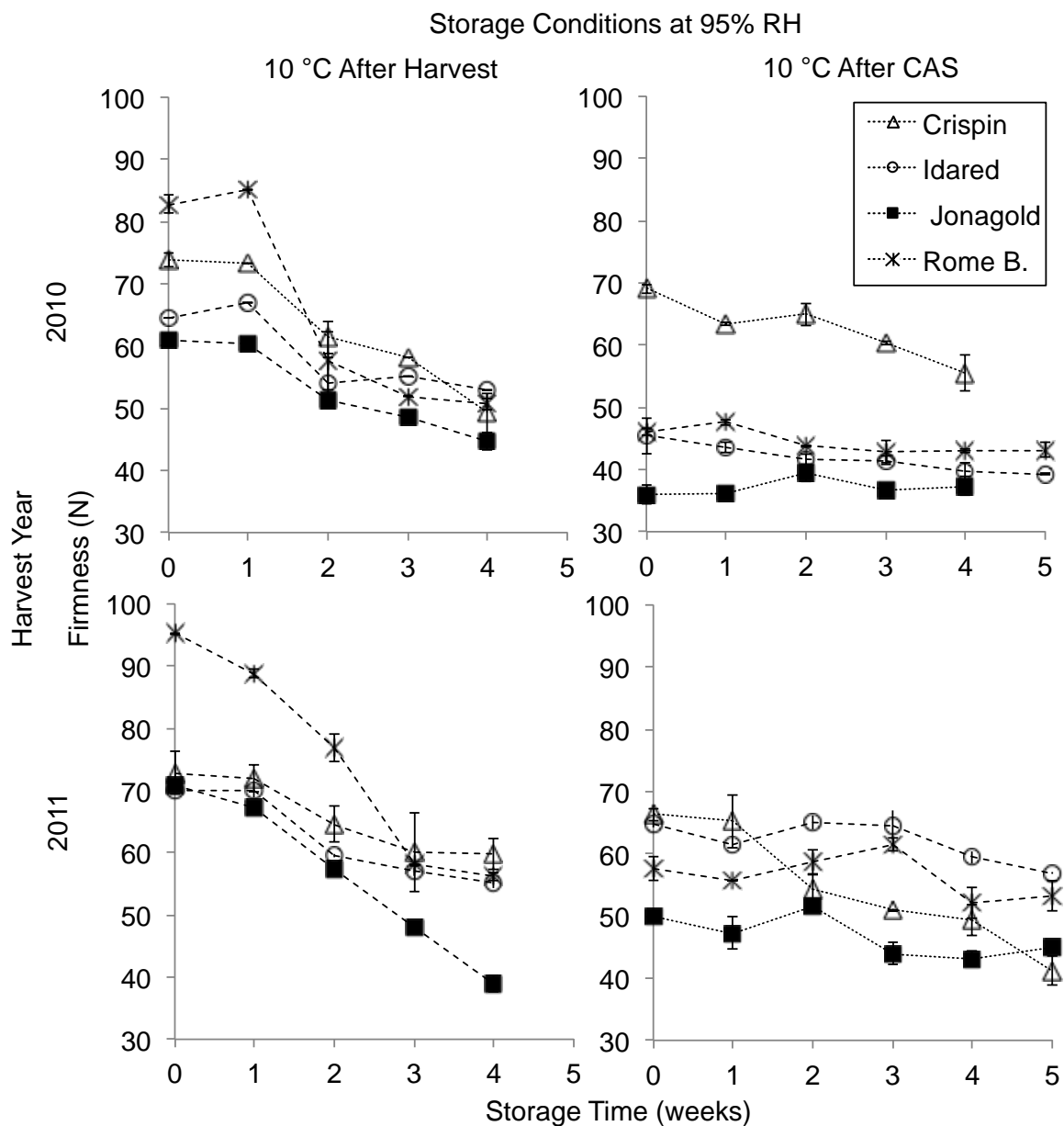


Figure 5.1 – Firmness of apples stored at 95% relative humidity (RH) at 10 °C for 4 or 5 weeks immediately after harvest and after coming out of controlled atmosphere storage (CAS) – 1-4 °C, 1-3% O₂ and 1-4% CO₂ for 7-10 months –, respectively, over 2 harvest years.

Our results are in agreement with previous literature on ripening indicator trends for both fresh and CA stored fruit with firmness, TA, pH and SS ranging 88 – 39 N; 0.969 – 0.152%; 3.12 – 4.00 and 9.9 – 16.1 °Brix, respectively (La Belle, 1981; Drake and others, 1979; Massey Jr., 1989; Vanoli and others, 2009; Blanpied, 1990).

Applesauce Rheological Properties

USDA Consistency

USDA Consistency – sauce and free-liquid flow (Figure 5.2) – of applesauce made from CAS group was overall significantly different than that of FS, reflecting changes occurring in the composition of sauce with extended storage, apparently beneficial for some varieties while detrimental for others considering harvest year effects. Jonagold and Rome were the most consistent varieties, yielding product of similar sauce and free-liquid flow to applesauce made from FS apples at end of the storage period (sauce flow = 5.75 ± 0.28 and 4.63 ± 0.37 cm; and free-liquid flow = 0.98 ± 0.32 and 1.04 ± 0.56 cm, respectively); Idared produced optimal sauce in 2010 (sauce and free-liquid flow mostly ≤ 6.5 and 1 cm, respectively) but substandard sauce (free-liquid flow ≥ 2.28 cm) in 2011; while Crispin produced substandard sauce over both harvest years (free-liquid flow = 3.19 ± 0.79 cm).

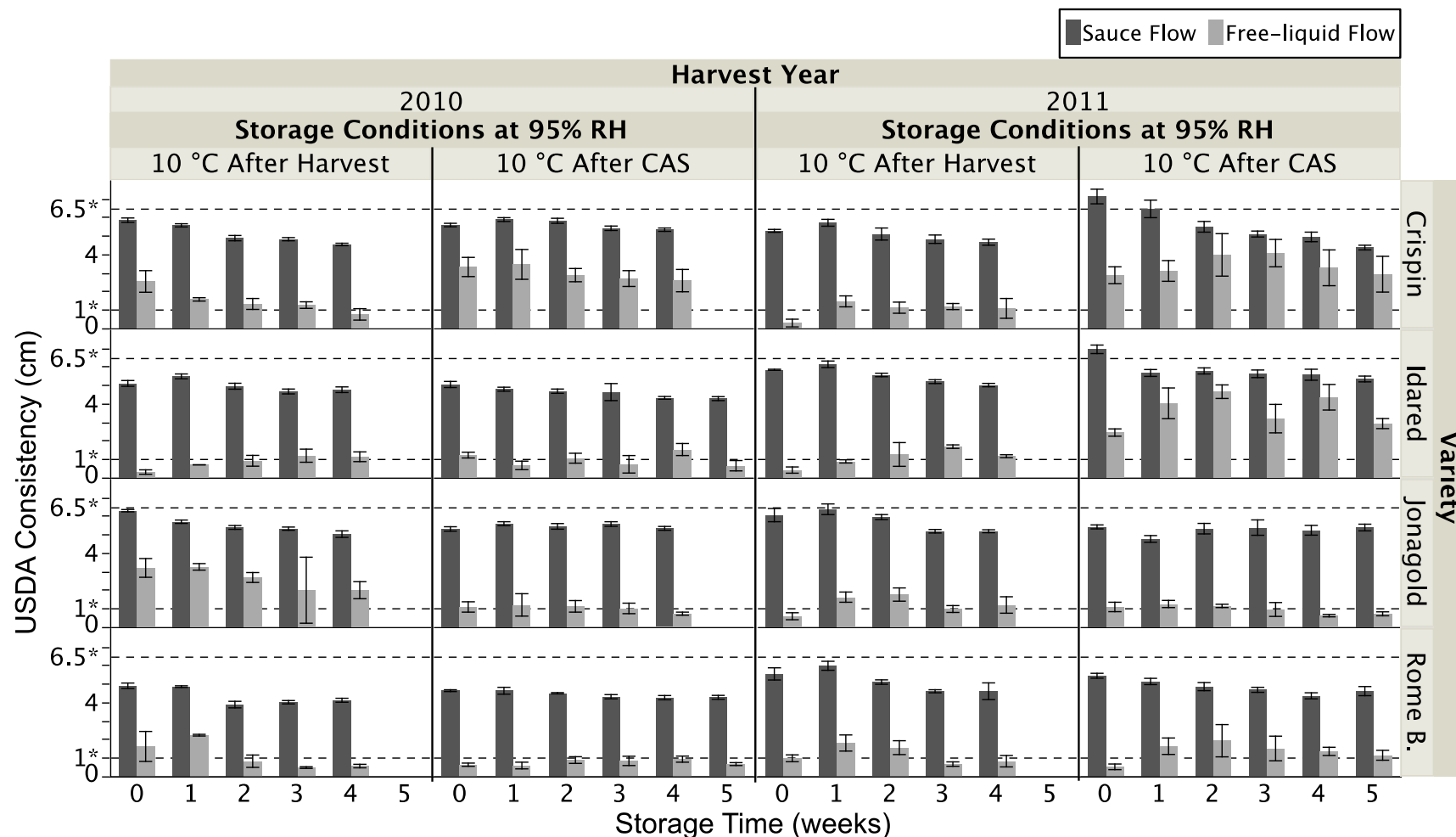


Figure 5.2 – USDA consistency (sauce and free-liquid flow) of applesauce made from apples stored at 95% relative humidity (RH) at 10 °C for 4 or 5 weeks immediately after harvest and after coming out of controlled atmosphere storage (CAS) – 1-4 °C, 1-3% O₂ and 1-4% CO₂ for 7-10 months –, respectively, over 2 harvest years. *6.5 and 1 cm are tracking parameters for applesauce consistency grading.

In a previous study (Chapter 4), our group observed the benefit of accelerated post-harvest fruit ripening at 10 °C and 95% RH for 3 weeks as a practice to improve rheological properties of applesauce made from freshly harvested apples (varieties: Cortland, Empire, Golden Delicious and McIntosh) due to acceleration of desirable ripening changes that affect the consistency of sauce. This effect was observed for sauce from FS but not CAS apples, probably because ripening changes do take place under CA storage conditions as demonstrated by fruit ripening parameters. The fact that CAS Crispin and Idared produced substandard sauce in this study could be indicative that ripening changes are not beneficial for all apple varieties, providing additional valuable information for sauce manufacturers for management of fruit blend over the processing year.

Drake and others (1979) reported the consistency of hot-break (steam-cooked) applesauce made from Golden Delicious apples stored under CA for 5 months to be 4.7 cm (sauce flow) and not different than that of sauce made from apples stored under cold storage – CS (1 °C) for the same period. Free-liquid flow was not reported likely due to its absence. Comparison between the studies is limited due to use of different methods for obtaining sauce as well as storage conditions and apple variety. It is important to mention, for instance, that in previous studies by our group with hot-break applesauce using CS apples (Chapter 2), applesauce consistency was significantly better (lower sauce and free-liquid flow) than that of cold-break applesauce in following studies for all varieties and storage conditions assessed (Chapters 3 and 4). Moreover, varietal and storage time effect on consistency was considerably less pronounced for hot-break

applesauce, indicating that cooking might have a significant effect on improving applesauce consistency regardless of storage condition.

Consistency Index and Yield Stress

Applesauce power law consistency index was strongly positively correlated with yield stress in both harvest years and storage conditions ($R^2 \geq 0.92$), and, therefore, only consistency index results are shown in Figure 5.3.

Yield stress and consistency index of sauces were significantly affected by storage condition, variety, harvest year, storage time and their interaction ($p \geq 0.05$), being significantly higher for sauce made from apples immediately out of CA storage than for sauce made from freshly-harvested fruit for all varieties in 2010 but only for Rome in 2011. Overall, both parameters significantly improved (higher values indicating stronger structure and greater resistance to flow) for both FS and CAS sauce with progress of storage time ($p \geq 0.05$) at 10 °C and 95% RH. Their values were negatively correlated to sauce USDA consistency, being overall higher for product showing lower sauce flow ($R^2 \geq 0.7$), and less so for product showing less free-liquid flow ($0.16 \leq R^2 \leq 0.56$ depending on storage condition and year). Ranges observed – 17–115 Pa and 13–123 Pa.s for yield stress and consistency index, respectively – were comparable to those found in previous applesauce rheological studies – 31–87 Pa and 7–50 Pa.s, respectively (Barbosa-Canovas and Peleg, 1983; Rao and others, 1986; Qiu and Rao, 1988; Shijvens and others, 1998), considering differences in assessment methods and procedures to obtain sauce.

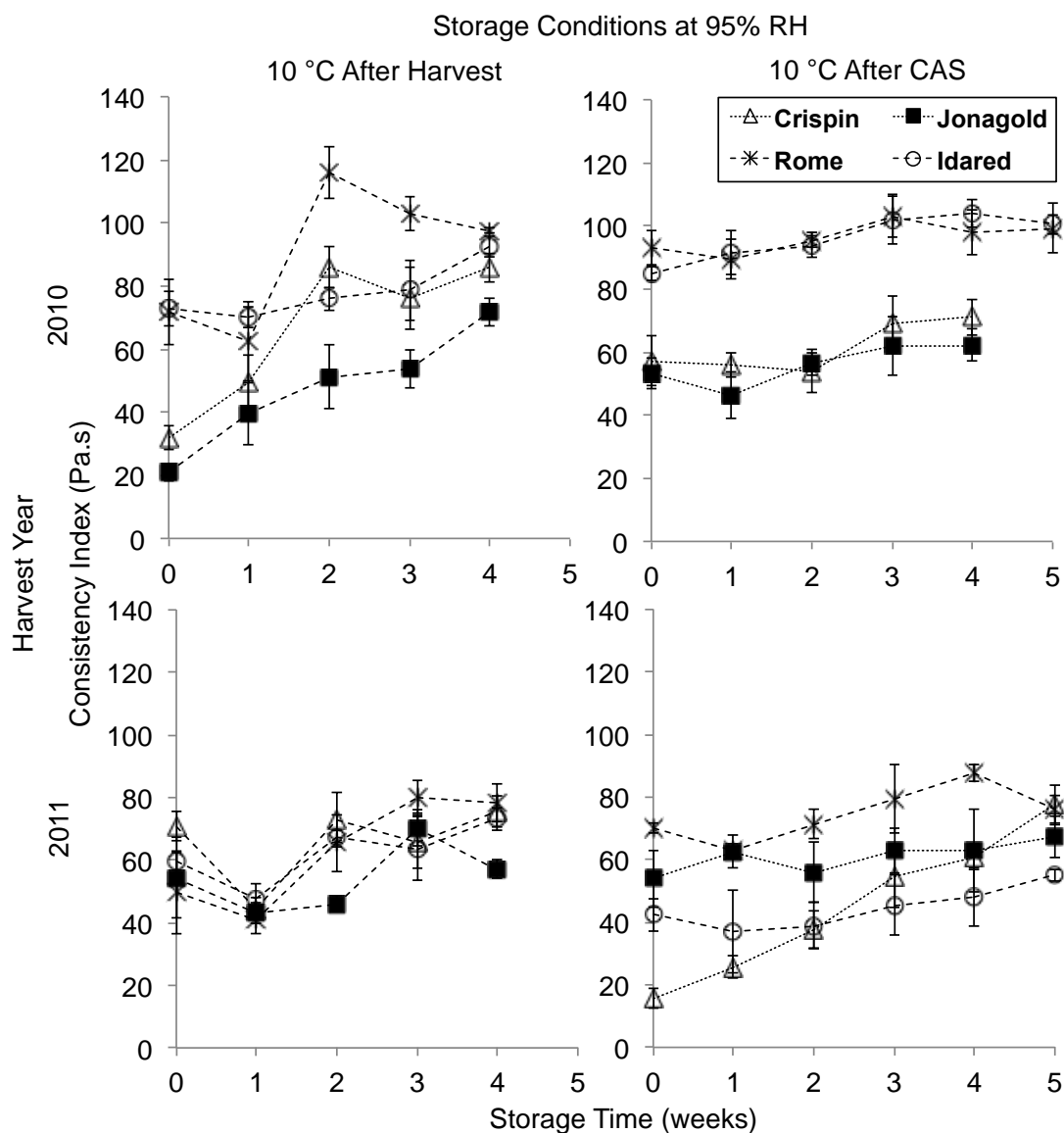


Figure 5.3 – Consistency index of applesauce made from apples stored at 95% relative humidity (RH) at 10°C for 4 or 5 weeks after harvest and after coming out of controlled atmosphere storage (CAS) – 1-4 °C, 1-3% O₂ and 1-4% CO₂ for 7-10 months –, respectively, over 2 harvest years.

Physical and Chemical Changes in Applesauce Composition

Differences in particle size distribution (PSD) of sauces with fruit storage are shown in Figure 5.4: most apple varieties coming out of CA produced sauce having PSD similar to sauce made from apples stored for 4 weeks at 10 °C and 95% RH, indicating that ripening changes affecting PSD of finished sauce also occur under CA storage, only at significantly lower rates. As a general trend for both groups, mean particle size (MPS) of sauces significantly decreased as distribution span (PSDS) increased proportionally ($p \leq 0.05$); ranging 1079 – 662 μm and 0.98 – 1.88, respectively, and having a marked varietal effect with progress of fruit storage as previously described by Mohr (1973 and 1989). PSDS was a significant factor for all rheological properties of sauce in this study ($p \leq 0.05$), being higher in sauce having higher yield stress and consistency index as well as lower sauce and free-liquid flow; while MSP was an additional significant factor for sauce free-liquid flow, being smaller in sauce showing less liquid separation ($p \leq 0.001$). Results support previous observations on the role of PSD on rheological properties of sauce (Chapters 2, 3 and 4). Our observations are in agreement with previous reports by Qiu and Rao (1988) on the negative correlation between average particle diameter and yield stress of applesauce.

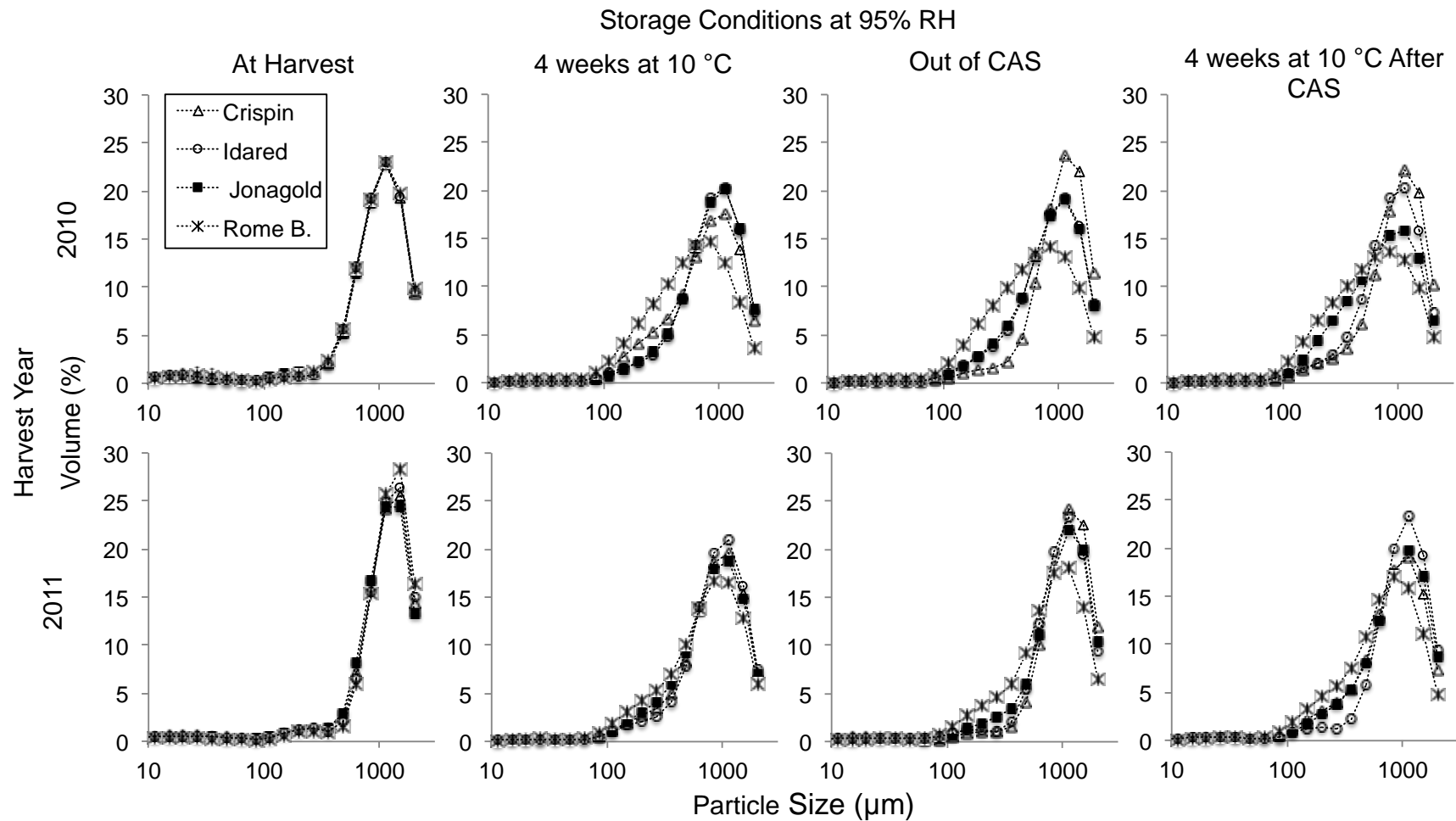


Figure 5.4 – Changes in particle size distribution of applesauce made from apples stored at 95% relative humidity (RH) at 10 °C for 4 weeks immediately after harvest and after coming out of controlled atmosphere storage (CAS) – 1-4 °C, 1-3% O₂ and 1-4% CO₂ for 7-10 months – over 2 harvest years.

Starch was not detectable ($\leq 0.1\%$) in CAS sauce and was only present in FS sauce during the first 3 weeks of fruit storage after which time it became negligible, having a significant beneficial effect ($p \leq 0.001$) on rheological parameters of sauce made from newly harvested apples (lower free-liquid flow). Levels in the beginning of the processing year (apple storage time = 0) in 2011 were significantly higher than in 2010 ($\leq 0.23\%$): Jonagold \geq Idared \geq Rome \geq Crispin (0.72, 0.43, 0.21, 0.13%, respectively). Absence of starch in CAS sauce further signals ripening changes occurring to apples under CA storage related to rapid starch degradation pattern after fruit harvest as reported in the literature (Brookfield and others, 1997; Belitz, 2009), while effect observed in rheological parameters of sauce are related to its functional properties as a thickening agent (Mason 2009).

Calcium in sauce made from CAS apples immediately out of CA was similar to that of sauce made from freshly-harvested apples, as expected since minerals are not consumed during fruit metabolism (Smock and Neubert, 1950). Levels in 2011 (13 – 68 ppm) were higher than 2010 (17 – 32 ppm). Results are in agreement with previous reports in the literature for apples, apple juice and pulp ranging 2-13 mg/100 g or 20-130 mg/L (ppm) on a fresh weight basis (Perring, 1974; Nour and others, 2010) and low range observed might have been due to analysis in serum. No significant effect was observed on applesauce rheological parameters as previously reported by Sams and Conway (1993) who studied the effect of calcium treatment of processing quality of apples, including applesauce. According to Pilgrim and others (1991), the calcium requirement for jellification in the presence of calcium averages 20 mg/g of low-

methoxylation pectin, indicating that it can take place at naturally calcium levels in applesauce depending on pectin chemical structure.

Total soluble pectin (TSP) content in sauce made from apples immediately out of CA storage was comparable to that of fresh apples in 2010, but overall significantly lower in 2011, with Crispin and Idared sauce showing the greatest losses over CA storage of fruit – Table 5.1. Levels observed (0.12 – 0.34%) are comparable with previous reports in the literature for pectin content in ripened or CA stored apples and in varietal applesauce (De Vries, 1981; Vanoli and others, 2009; Le Bourvellec and others, 2011).

TSP was overall higher in sauce showing lower sauce and free-liquid flow but it was not a significant factor for rheological properties of applesauce made from CA stored apples ($p \geq 0.05$). Applesauce alcohol insoluble residue – AIR, the residue after alcohol wash; which is composed of both the soluble and the insoluble fraction of pectins, other polysaccharide such as starch and a small amount of proteins (Ladaniya, 2008) – however, changed according to changes in TSP and starch content and was a significant factor for all rheological parameters of sauce, being higher for sauce showing less sauce and free-liquid flow, higher yield stress and consistency index ($p \leq 0.01$).

Results support previous research findings that AIR content is a stronger factor for rheological properties of cold-break applesauce rather than TSP (Chapter 3). Toldby and Wiley (1962) previously reported the negative correlation between pectin content and liquid-separation for hot-break applesauce. We observed similar results to the authors' when studying hot-break product (Chapter 2). Stronger effect of AIR than TSP for rheological properties of cold-break sauce could be related to its additional constituents, which can further prevent liquid separation in food systems (Stephen and Williams,

2006). Furthermore, AIR quantification is much simpler and involves considerably fewer investments in equipment than TSP and could potentially be tracked by sauce manufacturers for quality control of products. Differences in TSP and AIR between harvest years could be related to different weather conditions affecting fruit composition (Smock and Neubert, 1950).

Pectin degree of methoxylation (PDM) of sauces was overall stable over CA storage (Table 5.1) and, as for TSP, slight differences (increase or decrease) over storage could be attributed to de-polymerization of pectin fractions (insoluble to soluble) and de-esterification of the uronide carboxyl groups by enzymatic activity, notably of polygalacturonase and pectin methylesterase associated with fruit ripening (Knee and Bartley, 1981; Fischer, 1991; Van Burren, 1991). Some PDM results cannot be explained, however, and could be related to cumulative errors in the methodology assessment leading to large sample-to-sample variations and unexpected outcomes. PDM was higher for sauce having high sauce and liquid flow, but it was not a significant factor for rheological properties of sauce made from CA stored apples, a different behavior than observed in previous studies with cold-break sauce made from regular atmosphere stored apples (Chapters 3 and 4). Reasons for the different behavior can only be speculated. In that regard, it is important to mention that measurements of serum capillary viscosity of sauce made from regular atmosphere storage apples vs. from CA stored apples has shown that viscosity of CA applesauce serum is significantly lower, which could be related to changes in pectin molecular weight and conformation, in addition to changes in pectin content alone (Diaz and others, 2009).

Table 5.1 – Changes in alcohol insoluble residue (AIR), total soluble pectin (TSP) and pectin degree of methoxylation (PDM) of applesauce made from fresh apples and those stored at 95% relative humidity (RH) at 10 °C for 4 weeks after coming out of controlled atmosphere storage (CAS) – 1-4 °C, 1-3% O₂ and 1-4% CO₂ for 7-10 months – over 2 harvest years.

Parameter	Apple Variety	Harvest Year							
		2010				2011			
		At Harvest	Storage at 10 °C and 95% RH after CAS			At Harvest	Storage at 10 °C and 95% RH after CAS		
			Storage Time (weeks)				Storage Time (weeks)		
			0	2	4		0	2	4
AIR (%)	Crispin	1.84 ± 0.15 ^a	2.01 ± 0.29 ^a	2.16 ± 0.22 ^a	2.20 ± 0.14 ^a	2.48 ± 0.19 ^{a*}	2.00 ± 0.03 ^b	2.14 ± 0.01 ^b	2.42 ± 0.16 ^a
	Idared	2.86 ± 0.44 ^a	2.23 ± 0.20 ^b	2.51 ± 0.15 ^{ab}	2.60 ± 0.12 ^{ab}	3.09 ± 0.02 ^a	2.33 ± 0.44 ^b	1.85 ± 0.05 ^b	2.10 ± 0.12 ^b
	Jonagold	1.74 ± 0.17 ^b	2.15 ± 0.29 ^{ab}	2.52 ± 0.11 ^a	2.52 ± 0.24 ^a	2.17 ± 0.04 ^b	1.90 ± 0.07 ^b	2.76 ± 0.25 ^a	2.02 ± 0.15 ^b
	Rome B.	2.55 ± 0.27 ^a	2.26 ± 0.12 ^a	2.13 ± 0.01 ^a	2.24 ± 0.26 ^a	2.59 ± 0.20 ^a	1.88 ± 0.10 ^b	1.93 ± 0.14 ^b	1.98 ± 0.04 ^b
TSP (%)	Crispin	0.16 ± 0.01 ^c	0.25 ± 0.01 ^b	0.24 ± 0.01 ^b	0.36 ± 0.07 ^a	0.24 ± 0.01 ^{a*}	0.11 ± 0.02 ^c	0.09 ± 0.01 ^c	0.15 ± 0.01 ^b
	Idared	0.23 ± 0.04 ^a	0.18 ± 0.01 ^a	0.22 ± 0.04 ^a	0.22 ± 0.02 ^a	0.28 ± 0.04 ^a	0.16 ± 0.01 ^b	0.16 ± 0.02 ^b	0.13 ± 0.02 ^b
	Jonagold	0.21 ± 0.01 ^b	0.33 ± 0.03 ^a	0.33 ± 0.03 ^a	0.36 ± 0.05 ^a	0.29 ± 0.01 ^b	0.21 ± 0.03 ^c	0.35 ± 0.03 ^a	0.19 ± 0.01 ^c
	Rome B.	0.25 ± 0.01 ^a	0.21 ± 0.02 ^a	0.21 ± 0.01 ^a	0.22 ± 0.06 ^a	0.29 ± 0.04 ^a	0.16 ± 0.01 ^b	0.18 ± 0.02 ^b	0.10 ± 0.01 ^c
PDM (%)	Crispin	63.4 ± 9.5 ^a	49.8 ± 2.2 ^b	49.1 ± 0.2 ^b	53.6 ± 1.7 ^{ab}	55.4 ± 1.0 ^{a*}	45.9 ± 8.6 ^b	59.6 ± 5.0 ^b	49.1 ± 6.0 ^b
	Idared	63.8 ± 0.96 ^a	58.5 ± 0.5 ^b	58.9 ± 2.6 ^b	51.8 ± 3.6 ^c	41.4 ± 0.9 ^b	62.1 ± 6.2 ^{ab}	59.1 ± 13.8 ^{ab}	74.7 ± 22.1 ^a
	Jonagold	73.8 ± 5.1 ^a	53.2 ± 10.3 ^b	59.6 ± 0.1 ^{ab}	61.7 ± 9.3 ^{ab}	42.4 ± 3.6 ^b	53.5 ± 1.4 ^a	53.5 ± 1.4 ^a	49.3 ± 0.37 ^a
	Rome B.	41.8 ± 6.2 ^a	50.1 ± 7.6 ^a	58.8 ± 9.0 ^a	42.3 ± 11.5 ^a	45.4 ± 0.6 ^c	50.1 ± 7.7 ^c	76.8 ± 2.1 ^a	59.1 ± 1.0 ^b

*Apples stored at 1 °C and 95% RH for 3 months.

Conclusions

Ripening parameters signal that changes do occur to apples stored under CA that affect applesauce quality, although the practice extends fruit storage life significantly. Rheological properties of sauce made from CA stored apples were significantly different than sauce made from freshly-harvested apples due to changes in particle size distribution, starch and AIR, changes related to fruit ripening. Applesauce manufacturers can use AIR as a tracking parameter for starch and TSP content changes in sauce made from CA stored apples. The effect of particle size distribution on sauce rheological properties is of crucial importance for product made from CA stored apples when pectin levels might be reduced due to degradation under extended storage. The benefit of postharvest ripening practices, observed for sauce made from fresh fruit, was less marked or did not occur for sauce made from CAS apples, an important information for management of fruit blend for sauce manufacturers.

References

- Anthon, G. E., & Barrett, D. M. 2004. Comparison of three colorimetric reagents for the determination of methanol with alcohol oxidase. Application to the assay of pectin methylesterase. *J Agric Food Chem* 52: 3749–3753.
- Anthon GE, Barrett DM. 2008. Combined enzymatic and colorimetric method for determining the uronic acid and methylester content of pectin: Application to tomato products. *Food Chem* 110(1):239-47.
- Barbosa-Cánovas GV, Kokini JL, Ma L, Ibarz A. 1996. The rheology of semiliquid foods. *Adv Food Nutr Res* 39:1-69.
- Belitz HD, Grosch W, Schieberle P. 2009. *Food Chemistry*. 4th ed. New York: Springer. 1070 p.

- Blanpied, G.D. 1990. Controlled atmosphere storage of apples and pears. In: M. Calderon and R. Barkai-Golan (eds) Food preservation by modified atmospheres. CRC Press. Boca Raton FL, pp. 265-299.
- Brookfield P, Murphy P, Harker R, MacRae E. 1997. Starch degradation and starch pattern indices; interpretation and relationship to maturity. *Postharvest Biol.Technol* 11(1):23-30.
- Calvin L, Martin P. 2011. The U. S. Produce Industry and Labor: Facing the Future in a Global Economy, ERR-106, U.S. Department of Agriculture, Economic Research Service, November 2010. DIANE Publishing. 57p.
- Campanella OH, Peleg M. 1987. Determination of the yield stress of semi-liquid foods from squeezing flow data. *J Food Sci* 52(1):214-5.
- De Vries JA, Voragen AGJ, Rombouts FM, Pilnik W. 1981. Extraction and purification of pectins from Alcohol Insoluble Solids from ripe and unripe apples. *Carbohydr.Polym* 1(2):117-27.
- Diaz JV, Anthon GE, Barrett DM. 2009. Conformational Changes in Serum Pectins during Industrial Tomato Paste Production. *J Agric Food Chem* 57: 8453–8458.
- Drake SR, Nelson JW, Powers JR. 1979. The influence of controlled atmosphere storage and processing conditions on the quality of applesauce from Golden Delicious apples. *J Am Soc Hortic Sci* 104:68-70.
- FDA: 21CFR145.110 – Canned Applesauce [Internet]. Silver Spring, MD: U.S. Food and Drug Administration [Accessed 2012 Sep 17]. Available from: <http://www.accessdata.fda.gov/scripts/cdrh/cfdocs/cfcfr/CFRSearch.cfm?fr=145.110>.
- Fischer RL, Bennett A. 1991. Role of cell wall hydrolases in fruit ripening. *Annual review of plant biology* 42(1):675-703.
- Fischer M, Arrigoni E, Amadò R. 1994. Changes in the pectic substances of apples during development and postharvest ripening. Part 2: Analysis of the pectic fractions. *Carbohydr.Polym* 25(3):167-75.
- Gwanpua SG, Verlinden BE, Hertog MLATM, Bulens I, Van de Poel B, Van Impe J, Nicolaï BM, Geeraerd AH. 2012. Kinetic modeling of firmness breakdown in ‘Braeburn’ apples stored under different controlled atmosphere conditions. *Postharvest Biol Technol* 67(0):68-74.
- Kader AA. 1986. Biochemical and physiological basis for effects of controlled and modified atmospheres on fruits and vegetables. *Food Technol* 40(5):99-100, 102-104.

- Kidd F, West C. 1927. Gas storage of fruit. Dept Sci and Inst Res Food Inv Board Spec Rep 30.
- Knee M, Bartley IM. 1981. Composition and metabolism of cell-wall polysaccharides in ripening fruit. In: Recent Advances in Biochemistry of Fruits and Vegetables. New York: Academic Press. P 133-148.
- La Belle RL. 1981. Apple quality characteristics as related to various processed products. In: R. Teranishi and H. Barrera-Benitez. Quality of selected fruits and vegetables of North America. ACS Symposium Series. Washington: American Chemical Society. p. 61–76.
- Knee M, Bartley IM. 1981. Composition and metabolism of cell-wall polysaccharides in ripening fruit. In: Recent Advances in Biochemistry of Fruits and Vegetables. New York: Academic Press. P 133-148
- Ladaniya MS. 2008. Citrus fruit: biology, technology and evaluation. San Diego: Academic Press. 558p.
- Lidster PD, Sanford KH, McRae KB, Stark R. 1984. Apple juice quality and recovery from McIntosh apples stored in controlled atmospheres and air. *Can Inst Food Sci Technol J* 17: 86–91.
- Louis M, Massey JR. 1989. Harvesting, Storing and Handling Processing Apples. In: Downing DL. Processed apple products. New York: Van Nostrand Reinhold. P 215-238.
- Mason WR. 2009. Starch use in foods. In: BeMiller J, Whistler R. Starch. 3rd ed. San Diego: Academic Press. p 745-95.
- Massey LM, McLellan MR. 1985. Postharvest preprocessing temperature effects on the quality and particle size of finished apple sauce. *J Am Soc Hortic Sci* 110: 789–92.
- Massey Jr. LM. 1989. Harvesting, storing and handling processing apples. In: Downing DL. Processed apple products. New York: Van Nostrand Reinhold. P 31-51.
- McLellan MR, Blanpied GD, Massey LM. 1990. Harvest date and CA storage management effects on quality of processed apple slices. *J. Food Sci* 55(4):1046-8.
- MOHR WP. 1989. Influence of cultivar, fruit maturity and fruit anatomy on apple sauce particle size and texture. *Int J Food Sci Tech* 24(4):403-13.

- Ortega-Rivas E. 2012. Non-thermal food engineering operations. New York: Springer. 375 p.
- Patchen GO. 1971. *Storage of apples and pears*. Marketing Res. Rep. 924. U.S. Dept. of Agriculture.
- Perring MA. 1974. The chemical composition of apples. XI. An extraction technique suitable for the rapid determination of calcium, but not potassium and magnesium, in the fruit. *J Sci Food Agric* 25(3): 237–245.
- Pilgrim GW, Walter RH, Oakenfull DG. 1993. Jams, jellies and preserves. In: Walter RH. *The Chemistry and Technology of Pectin*. San Diego: Academic Press. p 23-50.
- Nour V, Trandafir I, Ionica ME. 2010. Compositional characteristics of fruits of several apple (*Malus domestica* Borkh.) cultivars. *Not Bot Hort Agrobot Cluj* 38 (3): 228-233.
- Qiu C, Rao MA. 1988. Role of pulp content and particle size in yield stress of apple sauce. *J Food Sci* 53(4):1165-1170.
- Rao MA, Cooley HJ, Nogueira JN, McLellan MR. 1986. Rheology of apple sauce: effect of apple cultivar, firmness, and processing parameters. *J Food Sci* 51(1):176-179.
- Rao MA. 2005. Rheological properties of fluid foods. In: Rao MA, Rizvi SSH, Datta AK. *Engineering properties of foods*. New York: CRC Press. p 41-98.
- Rocha AMCN, De Morais AMMB. 2000. Effects of controlled atmosphere on quality of minimally processed apple (cv. Jonagored). *J Food Process Preserv* 24(6):435-51.
- Sams CE, Conway WS. 1993. Postharvest calcium infiltration improves fresh and processing quality of apples. *Acta Hort* 326:123-130.
- Sahin S, Sumnu SG. 2008. *Physical properties of foods*. New York: Springer. 258 p.
- Sato ACK, Cunha RL. 2009. Effect of particle size on rheological properties of jaboticaba pulp. *J Food Eng* 91(4):566-570.
- Schijvens EPHM, Van Vliet T, Van Dijk C. 1998. Effect of Processing Conditions on the composition and rheological properties of applesauce. *J Texture Stud* 29(2):123-143.
- Siddiqui S, Brackmann A, Streif J, Bangerth F. 1996. Controlled atmosphere storage of apples: cell wall composition and fruit softening. *J Hortic Sci* 71(4):613-20.

- Smock RM, Neubert AM. 1950. Apples and apple products. New York: Interscience Publishers. P 486.
- Stephen AM. 1995. Food polysaccharides and their applications. CRC press, New York. 752 p.
- Toldby V, Willey R. 1962. Liquid-solids separation, a problem in processed applesauce. J Am Soc Hortic Sci 81:78-90.
- USDA: U.S. Apple Statistics [Internet]. National Agricultural Statistics Service, Cold Storage Annual Summary, various issues. Washington, D.C.: United States Department of Agriculture [Accessed 2012 Mar 31]. Available from: <http://usda.mannlib.cornell.edu/MannUsda/viewDocumentInfo.do?documentID=1825>.
- USDA: Grading Manual for Canned Applesauce [Internet]. Washington, D.C.: United States Department of Agriculture [Accessed 2009 Sep 19]. Available from: <http://www.usda.gov>.
- Usiak AMG, Bourne MC, Rao MA. 1995. Blanch temperature/time effects on rheological properties of applesauce. J.Food Sci. 60(6):1289-1291.
- Van Buren JP. 1991. Function of pectin in plant tissue structure and firmness. In: Walter RH. The chemistry and technology of pectin. San Diego: Academic Press. p 1-22.
- Vanoli M, Zerbini PE, Spinelli L, Torricelli A, Rizzolo A. 2009. Polyuronide content and correlation to optical properties measured by time-resolved reflectance spectroscopy in 'Jonagored' apples stored in normal and controlled atmosphere. Food Chem 115(4):1450-7.
- Watkins, C.B. 2003. Principles and practices of postharvest handling and stress. In: Apples: Crop Physiology, Production and Uses. Feree, D. and I.J. Warrington (eds) Chapt. 23, CAB Pub. pp. 585-614.
- Wiley RC, Binkley CR. 1989. Applesauce and other canned apple products. In: Downing DL. Processed apple products. New York: Van Nostrand Reinhold. P 215-23.

CHAPTER 6:
CONCLUSIONS, FUTURE WORK AND RECOMMENDATIONS

Summary of Findings

The present study generated information on the effect of apple variety, storage practices, post-harvest fruit ripening and harvest season on rheological properties of applesauce with a focus on product consistency. Information on physical and chemical parameters of applesauce composition affecting product rheology was also obtained and levels for achievement of optimal sauce consistency are reported for potential quality control purposes in industrial settings.

Considering varietal and growing season effects observed, general findings can be summarized:

- In 2009, the first harvest year studied, applesauce was obtained by cooking apples before sauce making, following an energy intensive and expensive hot-break process applied traditionally by applesauce manufacturers. Recently, applesauce lines have been evolving to a more efficient cold-break processing (studied in 2010 and 2011), when challenges with product consistency became more frequent. According to our observations, the hot break process yielded products of optimal consistency for all apple varieties over 8 months of cold storage indicating that the cooking effect on applesauce consistency is stronger than the effect of variety and storage time. This information can be of interest for adjustments to the processing line to minimize variation in product consistency considering both economical and product quality aspects.
- Rheological parameters of cold-break sauce, including consistency, significantly improved with apple post-harvest cold storage time (1-4 °C at 95% RH) with

optimal consistency being reached for most apple varieties after 2-3 months of fruit storage.

- Rheological parameters, including consistency, of cold-break sauce made from freshly harvested apples were significantly improved by accelerated post-harvest fruit ripening (storage at 10 °C for 2-3 weeks) prior to processing in the beginning of the harvest year.
- Rheological parameters, including consistency, of cold-break sauce made from controlled atmosphere (CA) stored apples, were significantly different than sauce made from freshly harvested apples. Accelerated post-harvest fruit ripening was not a beneficial practice for fruit coming out of CA storage because partial ripening had already occurred during the CA storage period affecting applesauce rheological properties, such as total starch loss and partial pectin degradation. As a result, mean particle size and particle size distribution stood out as the most important factors for the consistency of applesauce made from CA stored apples.
- Overall, applesauce rheological properties (consistency index, yield stress, USDA consistency) were affected by physical and chemical parameters of sauce – particle size distribution (mean particle size and particle size distribution span); starch, alcohol insoluble residue (AIR) and total soluble pectin (TSP) content; and pectin degree of methoxylation (PDM).
- Regarding sauce rheological properties, as an alternative or in addition to the USDA consistency analysis for grading purposes (according to manufacturers' discretion on claiming product grade or not), it is recommended that sauce

manufacturers track yield stress and/or consistency index for quality control purposes as a direct reading from a programmable rheometer.

- Regarding physical parameters, representation of particle size distribution (PSD) was successfully simplified by tracking mean particle size (MPS) and particle size distribution span (PSDS) which are direct measurements obtained from a laser diffraction particle size analyzer unit. Investment on such equipment is recommended for applesauce manufacturers for quality control of sauce consistency.
- Regarding chemical parameters, tracking starch, TSP content and PDM is not recommended as a routine quality control procedure due to methodology implementation limitations in the industrial setting related to time-consuming analyses, which are unlikely to help with decision-making at the fast pace required. Additionally, their effect was found to be limited: starch degrades quickly after harvest in apples (1-2 months under cold storage) and in finished products held at room temperature; while, if pectin content is low ($\sim \leq 0.2\%$), pectin degree of methylation does not seem to affect applesauce consistency, which was observed in particular with sauce made from CA stored apples and requires further studies. TSP was correlated with AIR content, and, therefore, implementation of the latter is recommended instead due to the methodology simplicity and few equipment requirements allowing for faster managerial decision-making. Furthermore, AIR content was observed to be a stronger factor for rheological properties of applesauce in comparison to TSP.

- Targeted levels for each parameter to achieve good consistency applesauce as well as full range of results for samples studied are provided as general recommendations:
 - Consistency index: optimal when ≥ 80 Pa.s (range: 13.5–380 Pa.s).
 - MPS: optimal when $\leq 800\mu\text{m}$ (range: 500–1200 μm).
 - PSDS: optimal when ≥ 1.5 (range: 0.9–2.25).
 - AIR: optimal when $\geq 2.5\%$ (range: 1.5 – 5.5%).
 - Starch: affects applesauce consistency when $\geq 0.25\%$ (range: 0-0.78%).
 - TSP: optimal when $\geq 0.25\%$ (range: 0.1–0.75%).
 - PDM: (optimal when $\leq 60\%$; range: 33–95%).

On a final note, it is important to mention that all findings herein presented are based on controlled studies modeling natural products. Specific information on physical and chemical parameters of applesauce for the achievement of optimal consistency for various formulated products requires optimization at the plant level by applesauce manufacturers.

Future Work

According to applesauce manufacturers, a blend of 3 to 7 apple varieties, out of a total of 20 or more commercially available for processing, are typically used as raw materials for sauce making in regular plant operation based on fruit availability, storage performance and expected contribution (often empirical knowledge) to particular desired attributes in the final product such as acidity, color, flavor, finish and consistency.

In the present study, only 10 different apple varieties were assessed. Most physical and chemical parameters affecting applesauce rheological properties were found to be highly variety-dependent, notably: post-harvest storage particle size distribution (affecting MPS and PSDS); AIR and TSP content; and PDM; while starch content was likely dependent on apple maturity during harvest and, as a result, was strongly affected by harvest year.

Thus, further studies on sauce made from varieties not contemplated in our experimental design would contribute to recommendations for manufacturers. In addition, all apples used in our study were supplied from farms located in New York State. The effect of apple growing region (East vs. West Coast) to physical and chemical parameters of sauce and its impact on product rheology is unknown.

Furthermore, although results provided in this study can potentially assist varietal blending by East Coast applesauce manufacturers targeting product consistency optimization, studies on “blending” as a processing procedure for improvement of applesauce rheological parameters were not carried out. As a result, recommendations on the usage of minimum and maximum proportions of optimal and challenging varieties for sauce consistency, respectively, in a given blend, cannot be made. Further research on the topic could be of additional assistance. Such research should contemplate the effect of fruit ripening in its scope.

Regarding novel technologies to further extend the storage life of apples, treatment of CA stored fruit with 1-methylcyclopropene (1-MCP) is a trending topic. The impact of the treatment on consistency of sauce made from CA stored apples is of additional interest for manufacturers.

Finally, advances in the quantification methodologies of chemical parameters (starch, TSP and PDM) affecting applesauce rheological properties are necessary allowing their faster and easier tracking and potential implementation at the industrial setting for product quality control purposes. In this regard, it is important to mention that we found considerable variability in PDM results and, therefore, further studies on PDM quantification in applesauce with more sample repetitions and apple varieties would be of interest to confirm its effect on rheological properties of sauce.

APPENDIX

A.1 – Excerpt of Applesauce Standards of Identity (FDA, 2012)

TITLE 21 - FOOD AND DRUGS CHAPTER I - FOOD AND DRUG
ADMINISTRATION, DEPARTMENT OF HEALTH AND HUMAN SERVICES
SUBCHAPTER B - FOOD FOR HUMAN CONSUMPTION PART 145 - CANNED
FRUITS subpart b - REQUIREMENTS FOR SPECIFIC STANDARDIZED CANNED
FRUITS

145.110 - Canned applesauce.

(a) Identity

(1) Definition. Canned applesauce is the food prepared from comminuted or chopped apples (*Malus domestica* Borkhausen), which may or may not be peeled and cored, and which may have added thereto one or more of the optional ingredients specified in paragraph (a)(2) of this section. The apple ingredient is heated and, in accordance with good manufacturing practices, bruised apple particles, peel, seed, core material, carpel tissue, and other coarse, hard, or extraneous materials are removed. The food is sealed in containers. It is so processed by heat, either before or after sealing, as to prevent spoilage. The soluble solids content, measured by refractometer and expressed as percent sucrose (degrees Brix) with correction for temperature to the equivalent at 20 °C (68 °F), is not less than 9 percent (exclusive of the solids of any added optional nutritive carbohydrate sweeteners) as determined by the method prescribed in Official Methods of Analysis of the Association of Official Analytical Chemists, 13th Ed. (1980), section 22.024, Soluble Solids by Refractometer in Fresh and Canned Fruits, Jams, Marmalades,

and Preserves Official First Action, which is incorporated by reference, but without correction for invert sugar or other substances.

(2) Optional ingredients. The following safe and suitable optional ingredients may be used:

- (i) Water.
- (ii) Apple juice.
- (iii) Salt.
- (iv) Any organic acid added for the purpose of acidification.
- (v) Nutritive carbohydrate sweeteners.
- (vi) Spices.
- (vii) Natural and artificial flavoring.
- (viii) Either of the following:
 - (a) Erythorbic acid or ascorbic acid as an antioxidant preservative in an amount not to exceed 150 parts per million; or
 - (b) Ascorbic acid (vitamin C) in a quantity such that the total vitamin C in each 113 g (4 ounces) by weight of the finished food amounts to 60 mg. This requirement will be deemed to have been met if a reasonable overage of the vitamin, within limits of good manufacturing practice, is present to insure that the required level is maintained throughout the expected shelf life of the food under customary conditions of distribution.
- (ix) Color additives in such quantity as to distinctly characterize the food unless such addition conceals damage or inferiority or makes the finished

food appear better or of greater value than it is.

(3) Nomenclature. The name of the food is applesauce. The name of the food shall include a declaration indicating the presence of any flavoring that characterizes the product as specified in 101.22 of this chapter and a declaration of any spice that characterizes the product.

If a nutritive sweetener as provided for in paragraph (a)(2)(v) of this section is added and the soluble solids content of the finished food is not less than 16.5 percent as determined by the method referred to in paragraph (a)(1) of this section, the name may include the word sweetened. If no such sweetener is added, the name may include the word unsweetened.

(4) Label declaration. Each of the ingredients used in the food shall be declared on the label as required by the applicable sections of parts 101 and 130 of this chapter. However, when ascorbic acid (vitamin C) is added as provided for in paragraph (a)(2)(viii)(b) of this section, after the application of heat to the apples, preservative labeling requirements do not apply.

Reference

FDA: 21CFR145.110 – Canned Applesauce [Internet]. Silver Spring, MD: U.S. Food and Drug Administration [Accessed 2012 Sep 17]. Available from: <http://www.accessdata.fda.gov/scripts/cdrh/cfdocs/cfcfr/CFRSearch.cfm?fr=145.110>.

A.2 – Excerpt of Grading Manual for Canned Applesauce (USDA, 2009)

Consistency measurement: suggested order of grading a sample unit.

All Styles

1. Gently and thoroughly mix contents of container(s) after taking the net weight, vacuum, and headspace. The applesauce should be as close to room temperature as possible (68-72°F; 20-22°C). Do not check consistency when the product temperature is over 80°F. In taking vacuum, warm product will register a lower vacuum than cold product.
2. Perform product examination using Inspection Aid No 105, Applesauce Tester Kit as shown in Figure 1 (Appendix III). Place the clean, dry cylinder directly over the center of the clean, dry USDA flow sheet 1, on a flat surface under approved lighting conditions, aligning the inside of the cylinder with the center circle.
3. Transfer the well-mixed sample to the cylinder so the applesauce will fill the cylinder level full.
4. Optionally, in the case of No. 10 containers, first transfer a well-mixed sample to a 600 ml beaker or other suitable container (No. 303 or No. 2-1/2 can) sufficient to fill the beaker or container before transferring the applesauce to the cylinder as previously described in step 3 of this procedure.
5. Remove any excess applesauce with a spatula or other suitable instrument by leveling off the top.

NOTE: Do not remove any free liquid that accumulates around the bottom of the cylinder.

6. With a smooth even motion, lift the cylinder straight up, allowing the applesauce to spread freely; let stand for one minute, then take reading immediately.

[...] (Item 7 refers to color grading)

8. Determine the consistency (extent of flow) by averaging the readings taken at the four quadrants of the flow sheet. (Readings are taken at the edge of the applesauce exclusive of any free liquid).

9. Determine the amount of free liquid, if any, by measuring the liquid from the edge of the applesauce at the four quadrants and averaging these measurements.

Regular (or Comminuted Style)

Grade A

The applesauce has good consistency. Good consistency means the product does not flow more than 6.5 cm (2.5 in); and there is not more than 0.7 cm (0.3 in.) of free liquid present. Canned applesauce that has a good consistency may be assigned a score of 18 to 20 points.

Grade B

The applesauce has reasonably good consistency. Reasonably good consistency means the product does not flow more than 8.5 cm (3.3 in); and there is not more than 1 cm (0.4 in) of free liquid present. Canned applesauce that has a reasonably good consistency may

be assigned a score of 16 or 17 points and should not be graded higher than U.S. Grade B regardless of the total score for the product.

Chunk (or Chunky Style)

Grade A

The applesauce has good consistency. Good consistency means the product does not flow more than 7.5 cm (2.95 in); and there is not more than a slight amount of free liquid present. Canned applesauce that has a good consistency may be assigned a score of 18 to 20 points.

Grade B

The applesauce has reasonably good consistency. Reasonably good consistency means the product does not flow more than 9.5 cm (3.75 in); and there is not more than a moderate amount of free liquid present. Canned applesauce that has a reasonably good consistency may be assigned a score of 16 or 17 points and should not be graded higher than U.S. Grade B regardless of the total score for the product.

Reference

USDA: Grading Manual for Canned Applesauce [Internet]. Washington, D.C.: United States Department of Agriculture [Accessed 2009 Sep 19]. Available from: <http://www.usda.gov>.